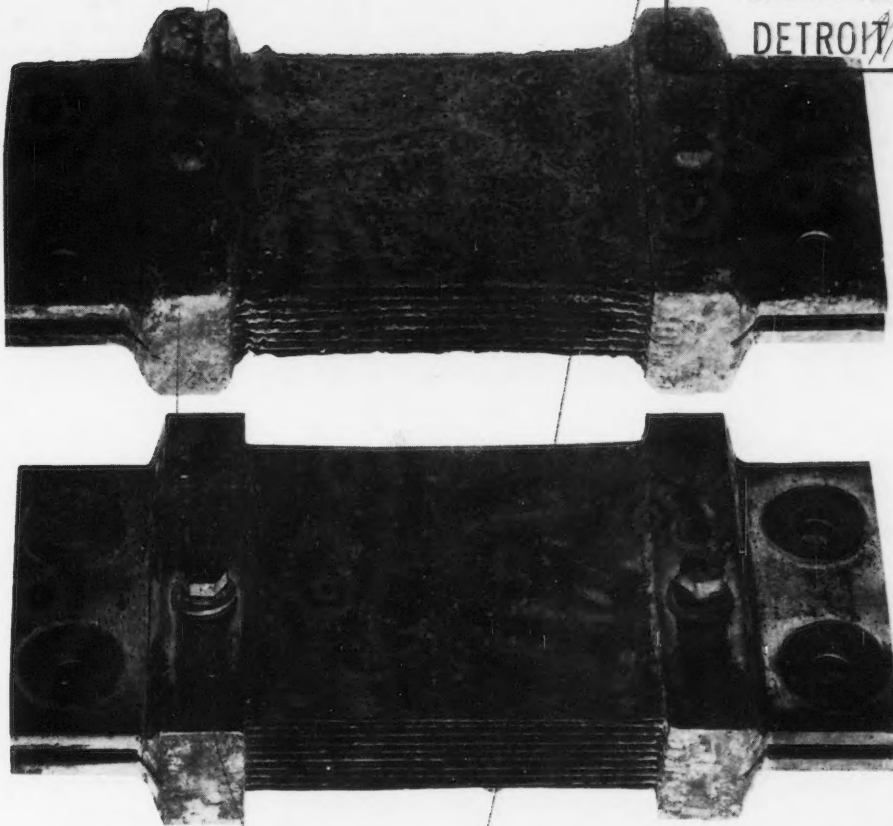


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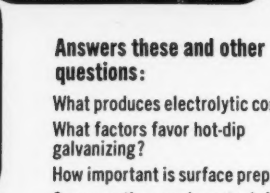
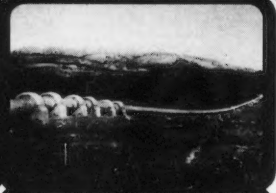
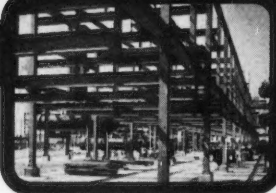
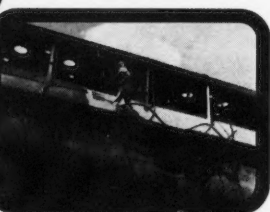
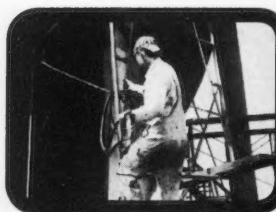
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Published monthly as its official journal, by the National Association of Corrosion Engineers, Inc., at Houston, Texas, U. S. A., as a permanent record of progress in corrosion control.

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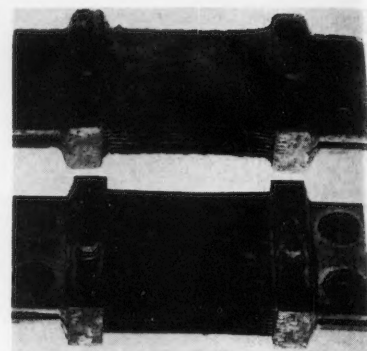
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### In January:

Precautionary Procedures in Chemical Cleaning. A Contribution to the Work of NACE Technical Unit Committee T-8A on Chemical Cleaning by Robert A. Stander, Chemical Service, Inc., Lafayette, La.

### In February:

Well Completion and Corrosion of High Pressure Gas Wells. A Status Report of NACE Task Group T-1B-1 by D. R. Fincher, Tidewater Oil Co., Houston; W. F. Oxford, Jr., Sun Oil Co., Beaumont; E. H. Sullivan, United Gas Corp., Shreveport, La.

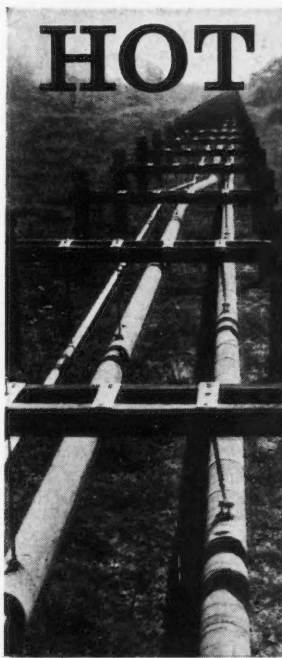
Chloride Stress Corrosion Cracking of the Austenitic Stainless Steels. A Contribution to the Work of NACE Group Committee T-8 via Task Group T-8A on Chemical Cleaning by J. P. Engle, G. L. Floyd and R. B. Rosene, Dow Chemical Co., Tulsa.

(Persons interested in the work of NACE technical committees are invited to write to J. F. Vander Henst, Jr., technical committee secretary, indicating the committee in which they are interested. A list of committees and their titles will be published in January CORROSION. A copy of the NACE Technical Committee Directory may be obtained on request.)

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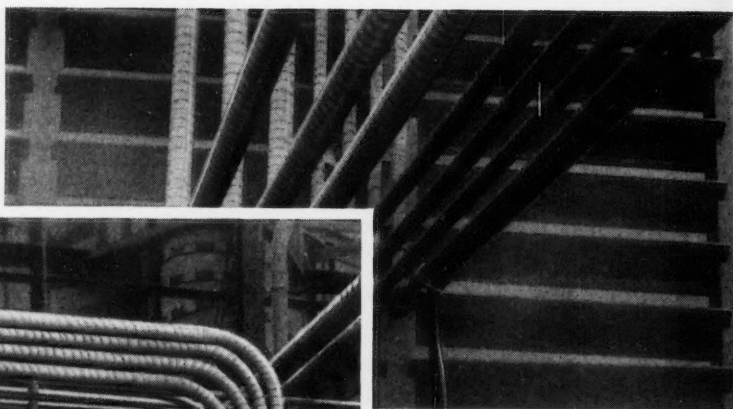
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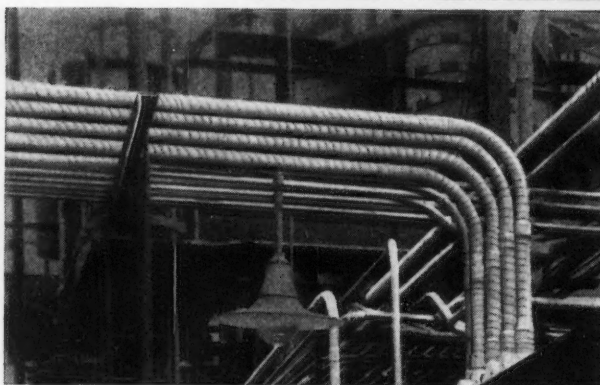
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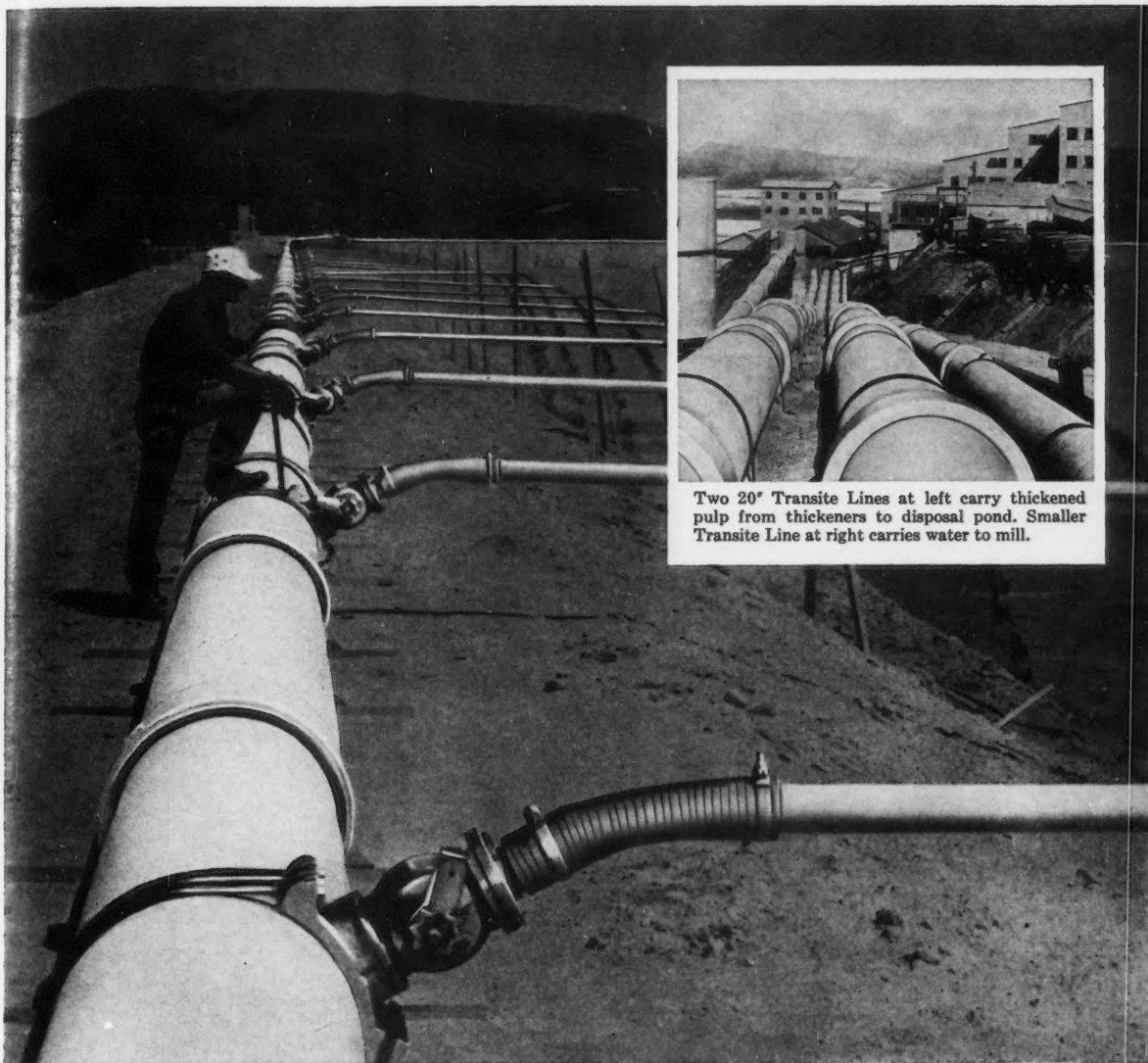
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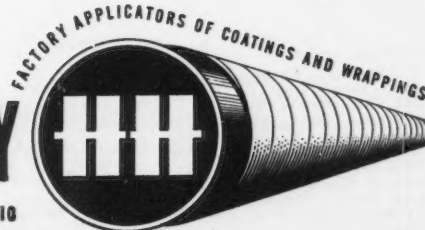
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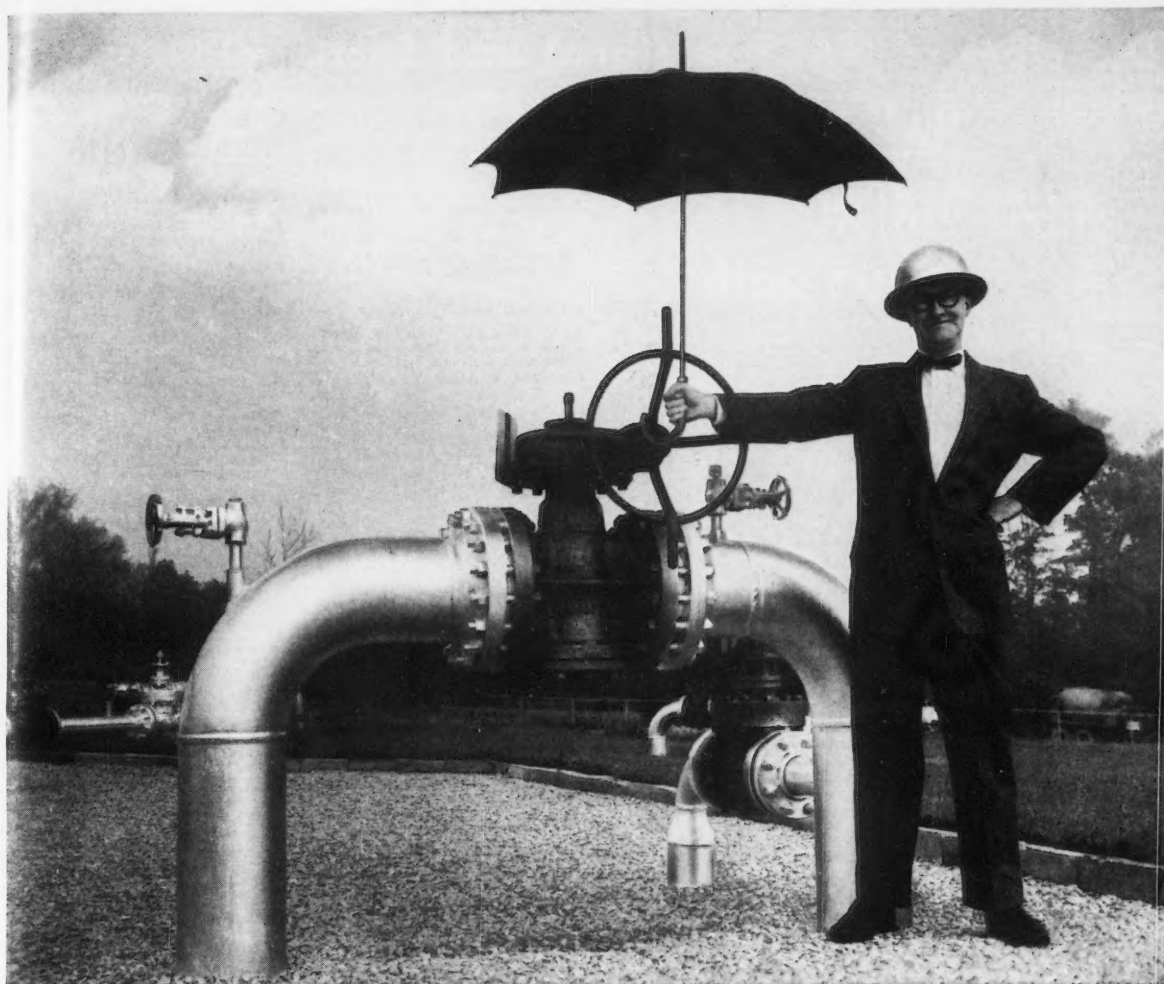
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## how CORROSION is indexed

- By Volumes annually in December issue
- In NACE Bibliographic Surveys of Corrosion
- In NACE Abstract Punch Card Service
- In 10-Year Index to CORROSION
- In Engineering Index
- In Applied Science and Technology Index

## Technical Articles Scheduled for January

High Temperature Corrosion Product Films on Aluminum by V. H. Troutner.

Mechanism and Kinetics of Aqueous Aluminum Corrosion.

Part 1—Role of the Corrosion Product Film in the Uniform Aqueous Corrosion of Aluminum by V. H. Troutner.

Part 2—Kinetics of Aqueous Aluminum Corrosion by R. L. Dillon.

Precautionary Procedures in Chemical Cleaning. A Contribution to the Work of NACE Technical Unit Committee T-8A on Chemical Cleaning by R. A. Stander.

Aluminum Cooling Towers and Their Treatment by A. J. Haygood and J. Dean Minford.

Corrosion of Steel Weldments by J. U. MacEwan and H. H. Yates.

Note on the Behavior of AZ63 Alloy and Magnesium 1% Mn Alloy in NaCl Electrolyte by J. H. Greenblatt and E. Zinck.

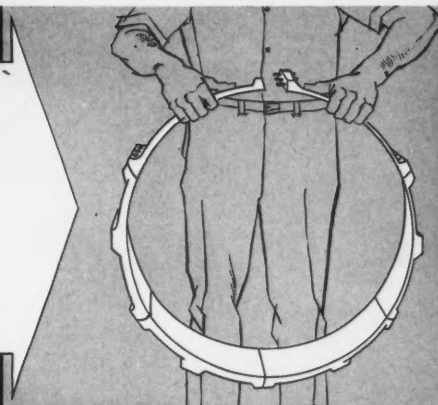
A Critical Analysis of Pitting Corrosion by Mars G. Fontana.

An Electrochemical Study of Pitting Corrosion in Stainless Steels, Part 1—Pit Growth, Part 2—Polarity Measurements by N. D. Greene and M. G. Fontana.

Deterioration of Wood by Marine Boring Organisms by Harry Hochman.

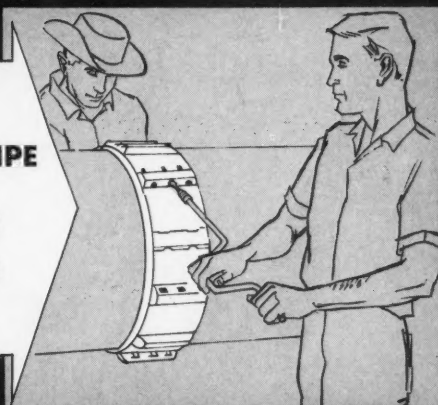


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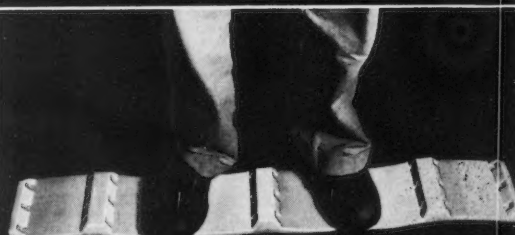
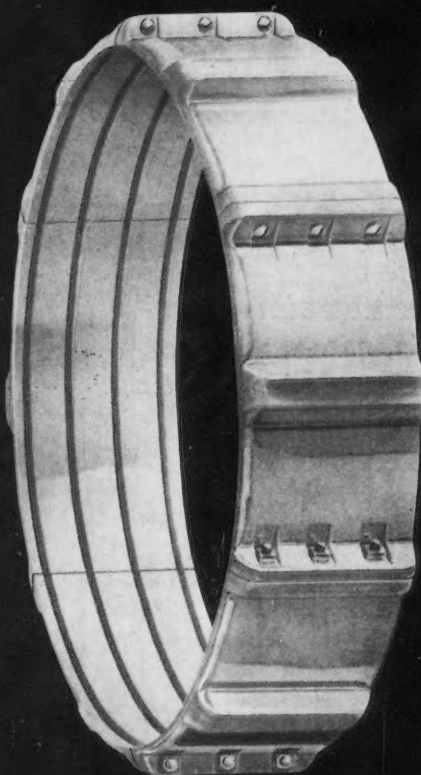
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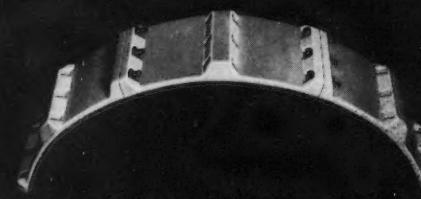
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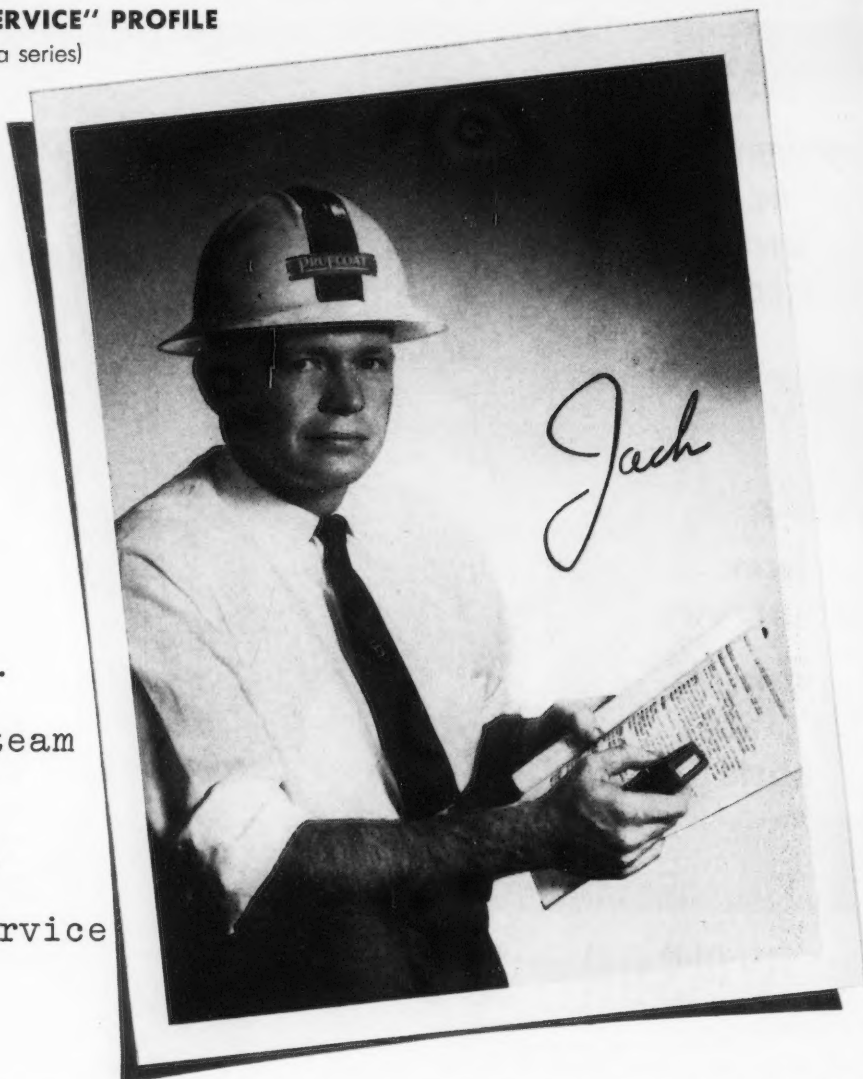


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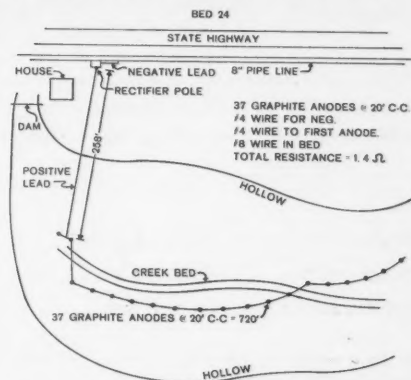
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## A Technical Note

# Effect of Increasing the Area of Iron to Zinc On the Rate of Reversal of Potential in a Zinc-Iron System\*

By HENRY L. SHULDENER<sup>(1)</sup> and LEO LEHRMAN<sup>(2)</sup>

### Introduction

IT HAS been assumed that the zinc coating of galvanized piping through which water flows protects the underlying iron electrochemically. In most instances the potential of the zinc is anodic to the iron. Schikorr<sup>1</sup> found, however, that under certain conditions, especially above 140 F, iron becomes anodic to zinc. Other authors showed later that the composition of the water, as well as higher temperatures, are the controlling factors for this "reversal of potential."<sup>2-5</sup> Recently Shuldener and Lehrman demonstrated that  $\text{HCO}_3^-$  has a very strong influence in causing reversal of potential of a zinc-iron system at 160 F with a consequent formation of rust<sup>6</sup> (see Figure 1).

In all of the previous work carried out on the reversal of potential, the area of zinc to iron was always constant. However, it is well known that galvanized pipe has flaws in the zinc coating

such as skips, flaws and pin holes that expose the iron underneath. Furthermore, threaded joints present varying areas of exposed iron. Inasmuch as the thickness of the zinc coating varies not only from pipe to pipe, but also on short lengths (6 inches and 14 inches) of the same pipe,<sup>7</sup> the amount of exposed iron after the zinc dissolves will vary. Therefore, experiments were carried out to determine what effect varying ratios of iron to zinc areas would have on the rate of reversal of potential as indicated by the appearance of rust.

### Experimental

The apparatus used and the experimental procedure was the same as used previously,<sup>6</sup> except that the amount of exposed iron of the test pieces, made by cutting through the galvanizing to the underlying iron, varied.

Table 1 shows the results of the experiments.

### Abstract

It is well known that under certain conditions iron becomes anodic to zinc in water systems. The experimental work carried out on the reversal of potential used a constant zinc to iron area. However, in galvanized pipes carrying water the area of iron exposed varies due to threaded joints, flaws in the zinc coating and solution of the zinc. As the ratio of iron to zinc area is increased it is shown that the reversal of potential is faster.

3.6.4

### Conclusion

The results show that for the same composition solution, reversal of potential was always faster when the ratio of iron to zinc area was increased.

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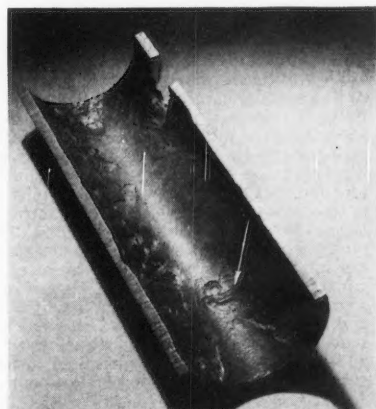


Figure 1—Cleaned section of galvanized wrought iron hot water pipe. Effect of reversal of potential is shown by extensive areas of galvanizing still present. Arrow points to galvanizing layer present up to edge of pit.

TABLE 1—Data from Experiments to Determine Effect of Fe-Zn Area on Rate of Reversal of Potential

Exp. No.	Type of Test Piece	Solution Used	Composition of Solution—(ppm)			pH	Time to Show Rust
			$\text{SiO}_2$	$\text{HCO}_3^-$	$\text{SO}_4^{=}$		
1 & 2	1 hole (2 mm dia.)	Catskill water + sodium silicate + sodium bicarbonate	10	14	11	8.5	None in 4 days
3 & 4	1 groove (3 mm wide)	Catskill water + sodium silicate + sodium bicarbonate	10	14	11	8.5	1 day
5 & 6	12 grooves (1 mm wide)	Catskill water	1.5	4	11	7.1	½ hour
10 & 11	1 groove (1 mm wide)	Catskill water	1.5	4	11	7.1	1 hour
12	12 grooves (1 mm wide)	Catskill water + sodium silicate + sodium bicarbonate + sodium sulfate	10	14	38	8.5	5½ hours
13	12 grooves (1 mm wide)	Catskill water + sodium silicate + sodium bicarbonate + sodium sulfate	10	14	38	8.5	3½ hours
20	1 groove (1 mm wide)	Catskill water + sodium silicate + sodium bicarbonate + sodium sulfate	10	14	38	8.5	9 hours
21	1 groove (1 mm wide)	Catskill water + sodium silicate + sodium bicarbonate + sodium sulfate	10	14	38	8.5	10 hours

# A Coating Evaluation Testing Program\*

By E. R. ALLEN, JR.

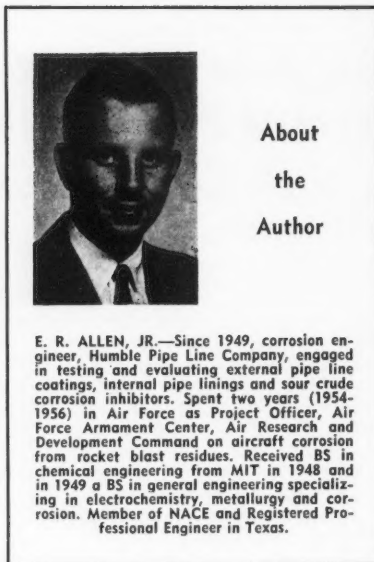
## Introduction

**H**UMBLE PIPE Line Company has been conducting a laboratory and field program for the past ten years to evaluate and test external pipeline coatings. At the time this program was started, the increased use and new developments in the coatings field made it obvious that some program was needed to learn more about the coating materials in common use and to establish some criteria by which new materials could be evaluated for possible application as pipeline coatings. New materials, which are now being offered more frequently for use as pipeline coatings, have made a coating testing and evaluation program even more desirable than ever before. This paper is presented to acquaint others with the approach of one company to this problem in the hopes that it will stimulate discussion of this important phase of coating work and, perhaps, encourage the publication of different approaches which others have used.

The first step in setting up this coating evaluation program was to determine just what constituted a good pipeline coating and what were its characteristics. The degree of protection obtained from a pipe coating was considered a function of its electrical resistance and of its freedom from holidays. The effective service life of a coating depends upon the inherent chemical stability of the component materials and upon the ability of the coating to withstand those destructive mechanical and chemical forces to which the buried pipe is subjected. During construction of a pipe line, the coating is subjected to stresses of impact, compression, flexing and bending at temperatures which sometimes range to both extremes. In underground service the coating must resist soil stress deformations and action of soil water, petroleum oils, and the effect, if any, of cathodic protection currents.

Many of the laboratory procedures used to study coating performance characteristics when this program was started did not supply the information required. There were several reasons for this. First, some of the methods used to test coating properties were subject to error. For example, in determining rate and amount of water sorption, all results obtained by the "nipple and can" method are open to question, since in a number of instances, water has been found to circumvent the sealing end cap. Immersion of small samples in water and measuring weight differences due to other causes such as leaching, oxidation, and volatilization of material components. Second, in some cases the property tested was of questionable significance. Thus, a tensile strength test on a molded specimen evaluates no coating performance characteristics even though it may test one prop-

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About  
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E. R. ALLEN, JR.—Since 1949, corrosion engineer, Humble Pipe Line Company, engaged in testing and evaluating external pipe line coatings, internal pipe linings and sour crude corrosion inhibitors. Spent two years (1954-1956) in Air Force as Project Officer, Air Force Armament Center, Air Research and Development Command on aircraft corrosion from rocket blast residues. Received BS in chemical engineering from MIT in 1948 and in 1949 a BS in general engineering specializing in electrochemistry, metallurgy and corrosion. Member of NACE and Registered Professional Engineer in Texas.

erty of a coating material affecting performance. Furthermore, the relationship between performance of a molded specimen and performance of a coated pipe is not known. This latter is the third and major shortcoming of laboratory work alone: the failure to correlate laboratory test results with service performance. While the requirements for a suitable system of precise coating evaluation can be met only in closely controlled laboratory type tests, laboratory results must be correlated with underground service performance.

Underground tests by themselves do not produce the desired information. Results of these tests alone are subject to many uncertainties and qualifications because the variable conditions which govern results can neither be controlled nor evaluated with any precision.

With this in view, the initial coating investigation was to consist of both field examinations of existing coated pipe lines and laboratory evaluation of the most commonly used coatings.

Field examinations were to be conducted on sections of pipe line representative of coatings and conditions selected for examination. The scheme of field examination was divided into six elements:

1. Summarizing details of coating application and history of pipe line
2. Determination of overall effectiveness of the coating
3. Traverse with the Pearson Holiday Locator
4. Inspection of the coating
5. Inspection of the pipe
6. Classifying destructiveness of the environment

## Abstract

A program to test and evaluate external pipeline coatings is described. When this program was started ten years ago, many methods and procedures then in use did not give all of the information desired. Field burial tests are lengthy and because of the variable conditions which govern results can neither be controlled nor evaluated with any precision. Only laboratory type tests are suitable for a closely controlled system of coating evaluation. Laboratory results must be correlated with field service performance.

This test program was in three phases: First, laboratory procedures and apparatus were developed. Second, commonly used coatings were tested and laboratory results correlated with field performance data. Third, new materials were tested and their performance compared with coatings in common use.

Laboratory and field test procedures and apparatus are described. A discussion on the application of test data to selection of pipeline coatings is included.

2.1.2

## Laboratory Test Procedures

Laboratory evaluation was to consist of three phases. First, to develop and devise laboratory procedures and apparatus suitable for evaluation of pipe coatings. Second, to evaluate commonly used coatings and to correlate laboratory evaluations with field performance data. Third, to test new materials with the techniques developed and evaluate their performance against coatings in common use. Coating characteristics to be tested in the laboratory were:

1. Electrical resistance
2. Rate and amount of water adsorption
3. Resistance to deformation under pressure
4. Resistance to cracking and spalling under impact
5. Resistance to cracking in bending
6. Adhesion to pipe metal
7. Deterioration in soil environment
8. Deterioration in petroleum oils
9. Effects of cathodic protection

In the laboratory program, the objectives were to develop methods of determining the performance characteristics of coatings under carefully controlled conditions. To do this, methods were devised so each test would determine one coating property under controlled, reproducible conditions. It was the intent, also, to determine these service performance characteristics on a complete coating structure as far as possible. As a result of this philosophy, tests were devised to measure resistance to soil stress penetrations at constant temperatures, to determine coating conductance under constant soil and moisture conditions and in the absence of soil stresses, resistance to impact, and other properties, all determined separately and under controlled conditions.

## Coating Samples

The service performance tests for coating samples are conducted on the actual coating structures applied to line pipe. A spinning rig is used to apply hot coatings



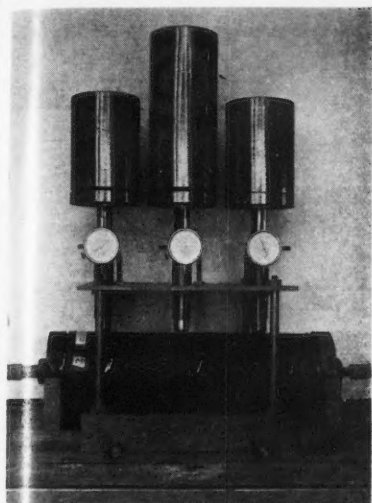


Figure 1—Laboratory deformation tester and coating sample. Sample was kept at constant temperature by circulating water.

to 4-inch pipe in a manner similar to that actually used in pipeline operations. Eighteen inch sleeves of 4-inch pipe are lined up on a mandrel to form an 18-foot length of 4-inch pipe to which a coating is applied. These coated sleeves are then separated by cutting the coating with a sharp knife at the end of each sleeve which results in 18-inch lengths of 4-inch pipe with a continuously applied coating. In general the coating samples were prepared by the laboratory. However, the newer types of thin coatings now being tested make it advisable to obtain coating samples from the suppliers because of the specialized techniques often involved.

The original laboratory tests for conventional hot applied coatings were based on the coating characteristic to be determined. A brief description of these tests follows.

#### Coating Deformation

Samples tested for coating deformation are coatings applied to 4-inch pipe. A  $\frac{1}{2}$ -inch radius hemispherical point with a 25-pound load is allowed to penetrate the coating at a constant temperature of 77 F to measure resistance of the coating to deformation under pressure. Test apparatus and a coating sample are shown in Figure 1. Depth of penetration and time elapsed are recorded until penetration ceases. Three weighted points are used for each coating. Penetration is reported as percentage of the total coating thickness to give a uniform basis for comparison despite variations in sample thickness. Results are shown as plots of the ratio of the depth of penetration to total coating thickness against elapsed time. Constant temperature is attained by circulating water at 77 F through the coated pipe.

A large number of coatings have been evaluated in this test. It is considered one of the more important tests on conventional hot-applied coatings. This test is used to determine the effect of the addition of glass fibre reinforcing inner wraps, the effect of an asbestos-felt outer wrap, and other variations in coating structure as well as differences in materials. The data shown in Figure 2 illustrate the type of information obtained in this test. Penetration curves are shown for four coating structures using the same enamel.

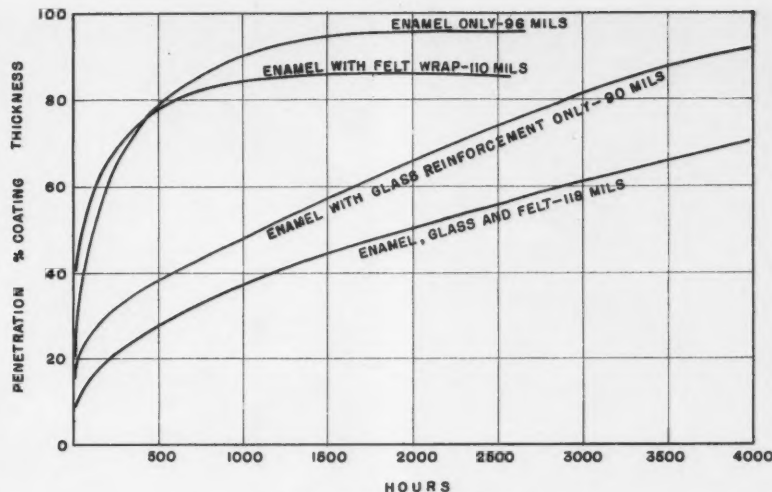


Figure 2—Coating deformation test.

tration curves are shown for four coating structures using the same enamel.

Data shown in the plot are not complete for the two coatings with glass reinforcement because deformation would continue until the curves flattened out. These data were presented to illustrate the variations which occur in rates of penetration. Curves shown for the enamel and enamel with felt coating are complete and show the manner in which deformation eventually ceases.

Determination of ultimate resistance to penetration is the coating property of major importance in this test; however, the rate of penetration is also determined and is significant, particularly the rate in the first few hours because it is during this period that a coating is subjected to much stress from handling during line construction. Construction is understood to include lowering and backfilling during which coatings are subjected to many uneven, intense pressures. There is a great difference in the initial penetration rates for different coating materials and structures. Materials whose resistance to deformation is inferior stand out quickly in this test.

The loading conditions and temperatures used were selected by experimentation as the most satisfactory. They cannot be varied if comparative results are desired but there is generally correlation with similar penetration tests made by others. This particular test procedure is applicable primarily to the hot-applied coatings which have a thickness of 85 mils or more. The procedure is not very satisfactory in testing thin coatings in the 5 to 25 mil range.

#### Effect of Cathodic Protection

Two tests have been set-up to determine the effects of cathodic protection currents on pipe coatings. The first test has been in progress for four years and is shown in Figure 3. Coating specimens on 18-inch lengths of 4-inch pipe are used. Eight specimens of each coating are placed under test; a perfect and a damaged specimen of each coating is being tested under four different applied potentials. Sample troughs are wooden boxes with one trough for each applied potential. Both ends of each specimen protrude from the trough through watertight seals. The ends of the specimens are treated to prevent current leakage.

Backfill is a natural soil slurry with a resistivity of 500 ohm cm and is kept saturated with distilled water to eliminate any soil stress.

Pipe-to-soil potential of the specimens is controlled by the applied potential. In one trough, 4.5 volts is applied to the coatings, 2.0 volts in the second, 0.1 volt in the third, and no potential is applied to the fourth trough which is used as a control. Data collected in this test are pipe-to-soil potential, coating conductance, and current collected by each sample. Periodic visual examinations are made to detect coating damage. Evidence of coating damage is also obtained from the current and coating conductance measurements. Examination of coating damage is shown in Figure 4.

The purpose of this original cathodic protection test was twofold: to evaluate coating performance under cathodic protection and to obtain some laboratory data on the effects of cathodic protection on pipe coatings. It was recognized that this would be a long term test; a great variety of coatings could not be included without making the test unwieldy. With this in mind, a limited number of coatings was selected which was believed to be representative of the different types of coating materials and coating structures in common use.

Coating performance in this test is based not only on the effect of current flow and resistance of the coating to blistering and lifting by hydrogen gas but also upon the electrical resistance of the coating and the moisture transmission rate of the coating. Although the effect of an applied potential is an additional factor to evaluate, performance of the undamaged sample in this test is determined by the moisture transmission rate of the coating. Because these are important properties affecting the service performance of underground coatings, this test was expanded to include a larger number of coatings. Some modification in test procedures was made to speed up testing.

The second cathodic protection test has been in operation for over a year. In this test, tap water adjusted to 500 ohm cm is used instead of a soil backfill. This was done so damage could be visually detected without disturbing the coating by removing backfill. Only two impressed voltages are used. One box is adjusted

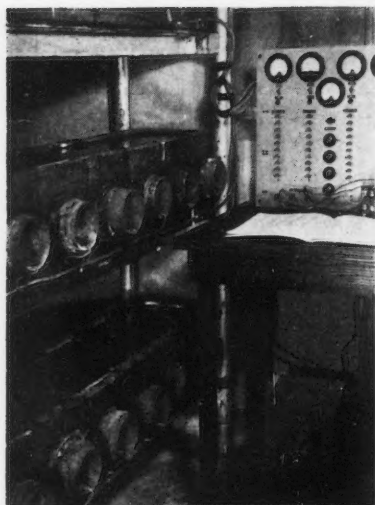


Figure 3—Cathodic current test boxes and test panel. Original test was made with soil backfill.

to have a pipe-to-solution potential of  $-1.00$  volt to a  $\text{Cu-CuSO}_4$  electrode. The second box has a potential of  $-4.5$  volts. One volt was selected for the lower voltage as it is in the range of normal cathodic protection practice and is below the hydrogen over-voltage and the damaging effect of rapid hydrogen gas evolution. The higher potential was selected as it gives more rapid results and is still within the range of potentials encountered in cathodic protection work. Only perfect samples have been placed in this test to date as it has been used to screen a large variety of experimental thin coatings in the 5 to 30 mil thickness range.

There is a danger in this type of test of having conditions too severe for some materials which would be good underground pipe coatings. In general, the test conditions are more severe than those usually encountered in actual soil conditions. This is because backfills have no shear strength to restrict the coatings and aid in preventing blistering. The test conditions would exist, however, on coatings on pipe lines in swampy areas or under water. Coating samples immersed in water and current data are shown in Figures 5 and 6.

The higher voltage test box has been helpful in eliminating unsatisfactory coatings. Both perfect and intentionally damaged samples indicate that the applied potential does not cause extensive coating damage until some current begins to flow. Coating damage has occurred primarily on those coatings which had initially high conductivity or holidays. This is particularly true for the thin coatings. Coatings with low initial conductance, with a few exceptions, have been unaffected by potential for over a year. In a few instances, coatings with low initial conductance have shown a rapid increase in conductance and rapid coating failure. This type of performance is believed to be typical of a coating with a high moisture transmission rate. It is significant that both the low potential and high potential samples of these particular coatings failed rapidly.

Testing of perfect coating samples eliminates only obviously unsatisfactory materials. Once current begins to be collected, there is considerable vari-

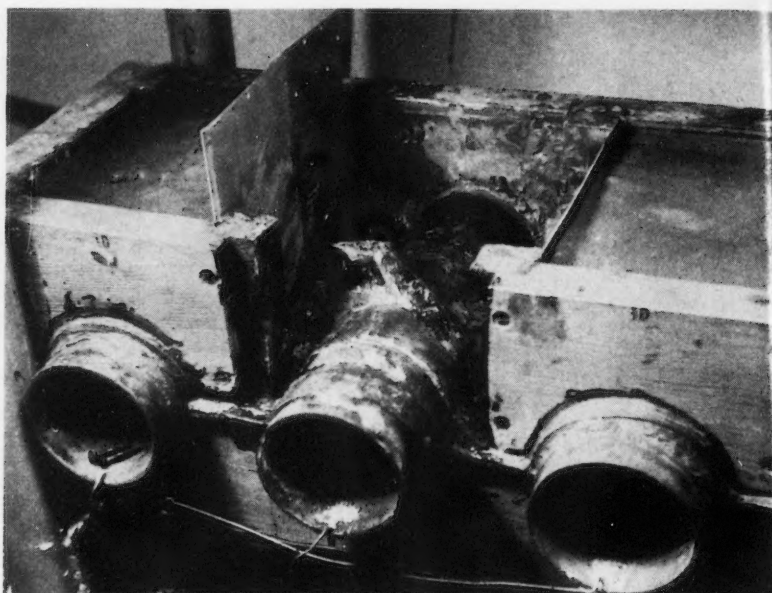


Figure 4—Cathodic current test box opened for inspection. Note large blister which occurred along faulty lap after two years at  $-4.5$  volts (pipe-to-soil potential).

ation in the ability of coatings to withstand further damage by hydrogen gas causing blisters and the high pH products formed. No coating should be considered to be satisfactory in its ability to withstand damage by cathodic protection until the performance of an intentionally damaged sample has been determined.

#### Soil Stress Deformation Test

In a test to determine the resistance of coatings to deformation by soil stress, coated samples of 4-inch pipe, Figure 7, are rigidly mounted in a test box which is filled with a gumbo clay soil. The soil is alternately wet and dried to produce soil stress pressures through the swelling and contraction of the soil. Appearance of the dried soil is shown in Figure 8. Reproducible wet and dry cycles are obtained by embedding soil moisture blocks in the soil. The soil is wet and dried to the same moisture percentage for every cycle. Drying is accomplished by air heated to about  $100^\circ\text{F}$  which is blown through a passage formed by hollow concrete blocks in the bottom of the soil box. Heated air is also distributed over the surface of the soil. The soil is wetted by spraying water from fog nozzles.

In operation over a year this test was initiated to evaluate a large number of thin coatings. Duplicate samples are tested, and coating conductance measurements are made for each sample. To date, no significant results have been obtained as the coatings originally placed under test are ones which would be expected to have good resistance to soil stress deformation. However, tests of this type have been conducted by others and have demonstrated severe soil stress damage on unsatisfactory coatings.

#### Soil Bacteria Test

As soil bacteria is an important factor in coating performance in certain parts of the country, this test is included in the program. The backfill used consists of top soil, peat moss, manure, agricul-

tural lime, and ammonium sulfate fertilizer. The soil is kept moist all of the time and is supposed to support rapid bacteria growth, particularly at temperatures above  $70^\circ\text{F}$ .

Coating samples on 4-inch pipe are buried in an outside pit with the special backfill to promote rapid bacterial growth. A lead is connected to each pipe sample for measuring coating conductance. Coating performance is determined by periodic visual inspections and by coating conductance measurements.

In this test, the sulfate reducing bacteria which enter into certain corrosion reactions are not significant, but rather the micro organisms of the type which exist in well aerated soils and are a factor in the deterioration of certain road surfaces. This test has not been in progress long enough for its performance to be evaluated.

The tests which have been described usually require six months to several years for completion. Also, these are tests to determine the underground service performance of coatings. Included in the test program is a series of laboratory tests to determine the resistance of various materials and coating structures to damage during pipeline construction. A brief description of these tests follows.

#### Enamel-Metal Adhesion

One factor in the ability of a pipeline coating to resist construction damage is the strength of the enamel-metal bond. There is also evidence that coating bond strength affects the underground performance of pipeline coatings, particularly if there is a poor bond.

In this laboratory impact-adhesion test, Figure 9, the average impact for failure of an enamel-primer combination is determined. Two primed metal discs, Figure 10, are bonded with molten enamel. After the enamel has cooled to testing temperature, the minimum impact necessary to cause failure of the bond is determined. One plate is suspended from a rigid bracket. A rod with a stop at the lower end is suspended



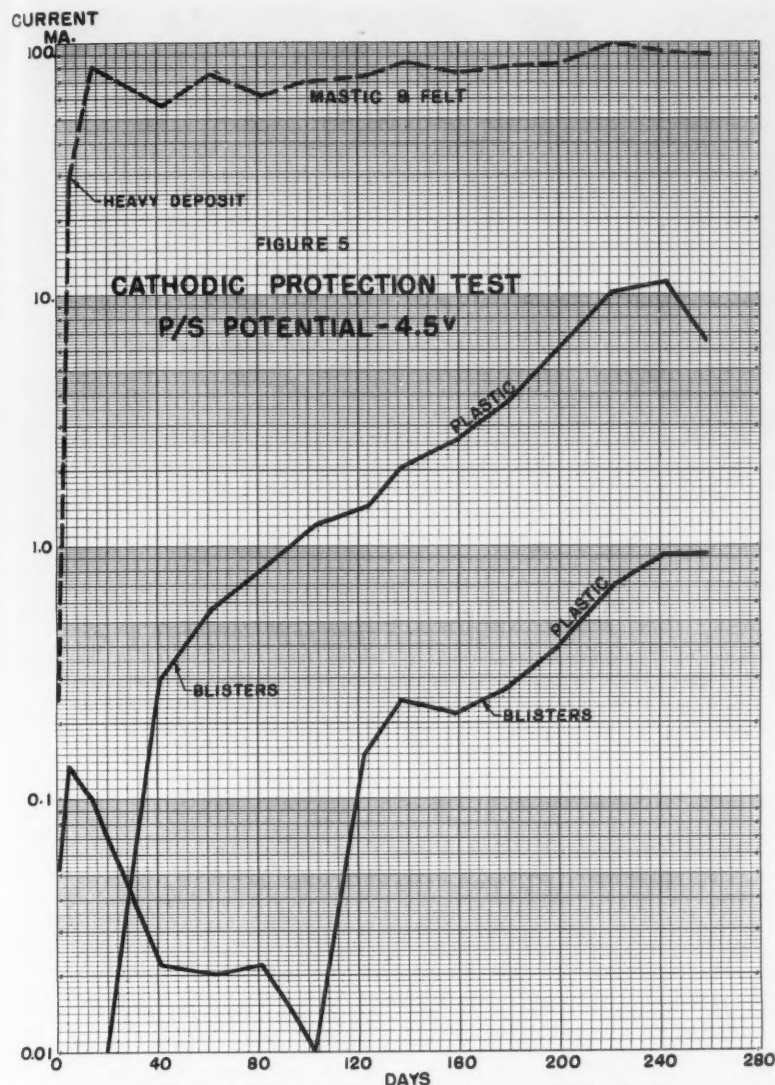


Figure 5—Cathodic protection test with pipe-to-solution potential of  $-4.5$  volts.

from the other bonded plate. A one-pound weight is allowed to fall freely from various heights down the rod to strike the stop, producing an impact tending to break the bond. At least ten samples are tested. Once the minimum impact is determined by successive drops of increasing height on the first one or two samples, the minimum impact for succeeding samples is found in two or three drops by starting slightly below the indicated height and working up.

Data are reported as the average impact for failure of the enamel-primer combination in foot-pounds per square inch. Observations made of the type of failures which occur are equally as important although they are difficult to state quantitatively. Some failures occur as parting of the primer-enamel bond, some as failure of the primer layer or primer-metal bond, and some as failures in the enamel layer.

Included in the data shown in Table 1 are the maximum and minimum values measured. Specific materials are shown as A, B, and C. In general, it was found that coal tar enamels fail at lower im-

pacts than asphalt enamels. However, failure of the coal tar in every case has occurred as parting of the enamel layer itself indicating that the adhesive strength of the enamel-metal bond is greater than the cohesive strength of the enamel. Asphalt enamels exhibit two to three times the overall impact adhesive strength of coal tars; however, failure of the asphalts usually occurs as parting of the primer-enamel bond or as failure of the primer layer or primer-metal bond. Of particular interest in the data shown are a coal-tar enamel with an asphalt primer and two asphalt enamels with coal tar primers. In these three cases, the strength of the enamel-metal bond is considerably less than when the matching primer was used.

#### Impact Damage of Pipe Coatings

Resistance of pipeline coatings to impact damage was tested with a Guillotine type apparatus in which a  $\frac{1}{2}$ -inch radius spherical point with a 10-pound weight was dropped on the coating from increasing heights. Complete coating structures on 4-inch pipe were tested.

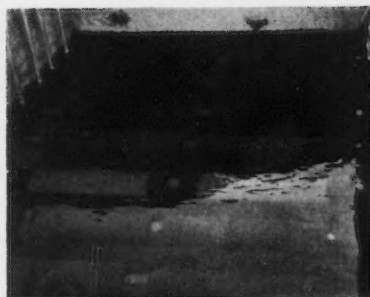


Figure 6—Cathodic current test. Samples were in 500 ohm-cm water.

TABLE 1—Impact Adhesion At 77 F.

	Ft-Lbs/Sq Inch
<b>Coal Tar Enamels With Coal Tar Primers:</b>	
Maximum.....	0.38
Minimum.....	0.11
A.....	0.36
<b>Asphalt Enamels With Asphalt Primers:</b>	
Maximum.....	3.75
Minimum.....	0.17
B.....	1.22
C.....	1.20
<b>Coal Tar Enamels With Asphalt Primers:</b>	
A.....	0.09
A.....	0.08
<b>Asphalt Enamels With Coal Tar Primers:</b>	
B.....	0.16
C.....	0.10

This test was discontinued because coating damage was difficult to detect when it occurred and because only gross differences in impact resistance could be determined.

#### Physical Properties and Effect of Heating

Pipeline enamels used for test purposes were usually obtained in drum lots. In order to correctly identify materials for comparison and to determine if the material tested was as specified, standard physical properties were determined according to ASTM procedures.

Physical properties determined for enamels were: softening point, needle penetration, specific gravity, percent insoluble in  $\text{CCl}_4$ , percent ash, moisture content, and loss on heating. Softening point and needle penetration after loss on heating were also determined.

ASTM test procedures used are described in Part II, ASTM Standards, 1944. All of the tests were run according to ASTM test specifications except loss on heating and moisture content. ASTM test D-95-40 was modified in determining moisture content to use a 200-gram sample and 500 ml of solvent because of the very low moisture content of the materials tested. ASTM test D-6-39-T conditions were modified to determine the effect of heating so that the sample was heated three hours at the manufacturer's maximum recommended pot temperature to more closely approximate field conditions.

#### Exposure to Oily Soil

In the early portion of the test program, attempts were made to evaluate the effect of crude oil soaked earth on



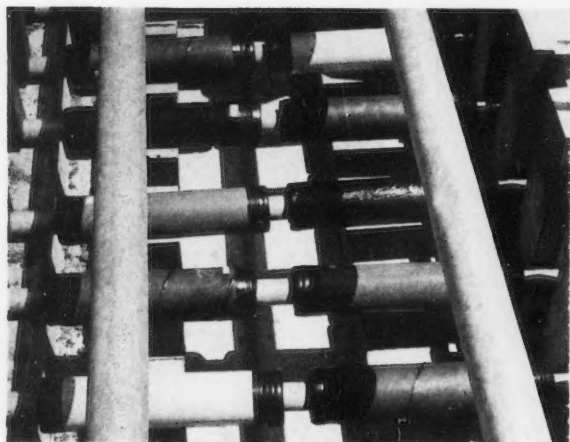


Figure 7—Samples in soil stress box. Four-inch pipes with coating applied were mounted on 2-inch pipe and clamped rigidly in box. Heated air for drying was circulated through galvanized duct above soil and through hollow concrete block duct in bottom.



Figure 8—Soil stress box. Note shrinking and cracking of dried soil.

pipeline coatings. Coatings on 4-inch pipe were placed in laboratory soil boxes and buried in moist soil saturated with crude oil. The boxes were sealed to prevent evaporation, and coating conductance was measured periodically.

Rapid failure was indicated for some coatings as shown in Table 2. Performance of the hot-applied felt wrapped coatings in this test agreed with field experience. However, in one series of tests on the early tape coatings, the laboratory results indicated good resistance to crude oil, but the tapes failed in less than six months in field tests in soils with just a trace of crude oil. The lack of soil stresses in this laboratory test gave erroneous results. The adhesive on the tapes softened, but there was no stress on the coating in the laboratory test to cause failure. At present, the effect of crude oil on coatings in the laboratory is determined by immersing coated pipe samples in oil and making visual examinations. These are rapid and severe test conditions; field installations are made of the more promising materials and result in more useful data.

The failure of laboratory results to agree with field tests experienced in this test indicates the necessity of always correlating a laboratory test with actual field performance. Once the limitations of a laboratory test are determined, it may be used with considerable reliability to predict the performance of new materials.

#### Field Test Procedures

Test procedures and techniques used in the field evaluation phase of the program are more or less standard ones familiar to most pipeline corrosion engineers. The objective is to determine the condition of the coating, the cause of any coating damage, and in general the effectiveness of the coating in a particular environment.

Every opportunity is taken during normal pipeline operations to examine coating which has been in service. Line repair and relocation work offer opportunities for coating examination; however, such operations do not give much selection in the coating and environment and do not result in repetitive examinations of the same line to accumulate a history of coating performance. Periodic examination of selected sections of

TABLE 2—Conductance of Pipeline Coatings in Oily Soil\*

DAYS	Enamel A and Felt	Enamel B and Felt	Enamel C and Felt	Enamel D Fiberglass and Felt
0.....	0.0074	0.0057	0.137	0.0069
13.....	0.0212	0.0143	0.274	0.0183
19.....	0.0228	0.0175	42.2	0.0217
25.....	0.032	0.024	262.0	0.0217
233.....	0.148	0.211	5940.0	38.8
417.....	2.01	0.964	6360.0	91.0
450.....	5.27	1.42	8900.0	255.0
481.....	5.27	1.39	7000.0	200.0
566.....	5.27	2.09	6550.0	236.0

\* Micromhos per square foot.

coated pipelines give the most useful performance data. If field examinations of selected lines are conducted, coatings and soil conditions of particular interest may be chosen.

Use of the Pearson Holiday Locator combined with visual examination of holidays is the basic method of determining the physical condition of the coating. Coating conductance measurements and cathodic protection current requirement tests are used to determine coating effectiveness. However, these latter two methods must be used with caution in evaluating a coating since they are influenced by soil conditions. Use of the Pearson Holiday Locator was described in "The Petroleum Engineer" in 1941 by Dr. J. M. Pearson. The method consists of impressing an interrupted direct current of audio frequency on the buried pipeline and locating points of current flow between the pipe and soil. Such points of current flow occur at coating holidays. Work with this instrument has demonstrated that all holidays in a generally good coating can be located positively and accurately. Distinction can be made between a generally good and a generally poor coating, and major holidays in a generally poor coating can be located.

Experience with a 276-mile 8-inch products line from Baytown to Dallas constructed during the summer of 1946 illustrates the information which can be obtained from a thorough field test program. Considerable money and effort were spent to obtain a good coating on this line. At the southern end, 127 miles were coated with coal tar enamel with a glass fibre reinforcing inner wrap and

an asbestos-felt outer wrap. This coating was applied at a central yard coating plant, and joints were hand-coated in the field. The northern section was coated over-the-ditch with an old type asphalt enamel using glass fibre reinforcing wrap and asbestos-felt outer wrap.

A Pearson Traverse of the 276-mile line was made in 1947-48 and again in 1954. Ten percent of the line was covered with a Pearson Holiday Locator by going over 5000 out of every 50,000 ft of line. An impartial selection of test sites was obtained by plotting test sections on the right-of-way alignment map irrespective of right-of-way conditions. About half of the holidays located were uncovered and inspected. As some indication of the size of the holiday is obtained in the Pearson technique, holidays were selected in an attempt to select some small, some intermediate, and some large breaks in the coating.

The results of this survey were surprising; a large number of holidays were found in spite of efforts made during construction to obtain an excellent coating job. This was true particularly for the survey made in 1947-48 shortly after the line was constructed.

Sixty-two holidays, an average of 5 per mile, were located in the inspected 10 percent of the southern half of this line with the yard applied coating. Examination of these holidays indicated that the major cause was construction damage to the coating. Approximately 40 percent of the holidays were caused by faulty application of field joints, the others by mechanical damage during construction. This damage was easily

determined in the inspection and was found to be caused by small tool cuts, gouging by dozer blades, penetration by skids, penetration by scrap metal, including welding rods, on the ditch bottom under the pipe. No effects of general deterioration or other breakdown of the coating were observed. Soil conditions in this section of line are considered severe because the soil includes gumbo clay of the Gulf Coast area and sections which change rapidly from sandy soil to tight clay.

The condition of the over-the-ditch coating on the northern half of this line was entirely different; 52 holidays for an average of 3.4 per mile were found. However, these were only the larger holidays as it was found that the coating was generally penetrated by soil clods. These penetrations were a result of lowering the hot, soft coating directly into the ditch and of soil stress deformation. Soil conditions in this section were also severe, varying from mixed sand and red clay to black clay and some soft limestone.

Rock and clod penetrations were the primary cause of over half the holidays in the northern section. Some of the penetrations were caused by lowering the hot coating into the ditch, but there were extensive penetrations on the top and sides of the pipe as well. Two random inspections were made where no holidays were indicated; extensive clod penetrations were found but had not completely penetrated the coating. Other causes of holidays were similar to those in the southern portion of the line: damage in handling, trench skid penetrations, and faulty application.

The second Pearson survey made in 1954 confirmed the results of the first. No general deterioration was found in the southern portion, only construction damage; the northern section was generally penetrated. Coating conductance measurements have also been made but have not given any comparative results because of varying soil conditions and techniques used.

The results of these surveys have been valuable because they give a good indication of the condition of the coating on this line and confirm the importance of certain practices and coating properties in obtaining good service life. Other field experience, limited to pipe sizes of 20 inches and under, proves that yard applied coatings are more satisfactory than over-the-ditch jobs. Also, insofar as differences in soil conditions can be taken into account, the resistance to soil stress penetration is a major factor in underground coating performance. Two of the holidays on the northern end, where considerable coating was dissolved from the pipe by gasoline coming from a weld sweat, emphasized the benefits of using coating materials which are unaffected by fluids carried in the line, other factors being equal.

The extensive rock and clod penetrations found in the northern section of this line gave excellent correlation with laboratory coating deformation test. The enamel, used in coating the northern section, proved to have poor resistance to deformation in the laboratory test. Construction of this line was completed before the coating test program was begun.

#### Summary of Test Program

In summary, it is felt that field examination techniques are well developed

and give satisfactory results. However, field data do not produce all the desired information. Results are often subject to uncertainties and qualifications because the variable conditions which govern results cannot be controlled.

Techniques for locating and examining holidays in a coating are well developed. Coating conductance measurements in the field have not been reliable. However, a great deal of work has been done on improving the methods and calculations involved in measuring coating conductance by NACE Committee T-2D. Conductance data alone cannot be used to evaluate a coating because too many unrelated factors affect results, but, when used in combination with Pearson surveys and soil resistivity measurements, a good evaluation of coating performance can be made.

Laboratory evaluation of coating performance has agreed in general with field experience. Ranking of materials in various laboratory service performance tests has agreed in most cases with performance of materials in the field. Coatings which have proved to be superior in actual field service have also been superior in laboratory performance tests. What may be of even more importance is that unsatisfactory coatings stand out very quickly in the laboratory tests.

There are two difficulties in the laboratory program which have not been solved satisfactorily. First, most laboratory results determine only a comparative ranking of materials in relation with other materials tested. No numerical values have been established for most coating properties to distinguish between acceptable and unacceptable performance. The second difficulty is that the most useful tests are the long term ones. Attempts have been made to predict laboratory service performance of coatings from accelerated tests or standard ASTM tests, but results have been poor. One property which has been approached along this line is the resistance to soil stress penetration. Attempts have been unsuccessful to correlate the maximum penetration with the rate of penetration in the early stages of the test or to develop a penetration index from the standard ASTM needle penetration test. Properties of a coating are due to the combination of materials in the actual coating structure on a pipe, and it has not been possible to use the specific properties of the individual components to predict performance.

There has been a change in the approach to laboratory testing in the past three years. This has come about because of new materials, mainly thin plastic coatings which are of increasing interest, and because of an evaluation of the testing program after several years.

Originally, primary interest was in the thick hot-applied conventional coatings in common use. The problem was approached by determining coating properties separately in closely controlled tests. An evaluation of results showed that the techniques, developed to measure every factor in coating performance separately, in some cases indicated relatively small differences in similar materials.

The most important tests proved to be lengthy, and the test program sometimes lagged because of unavailable testing facilities and because of the time needed to conduct all the separate tests

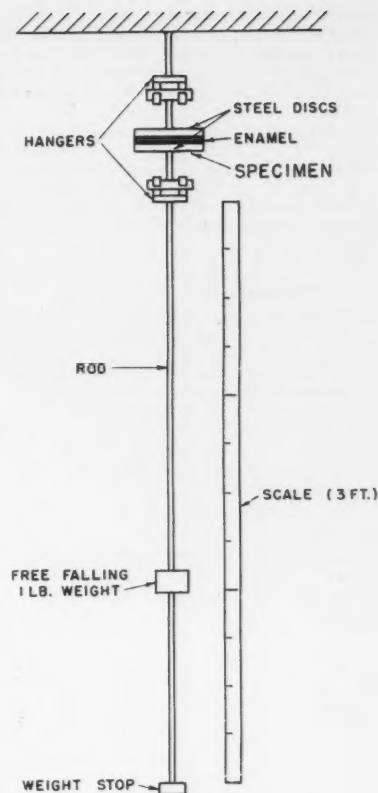


Figure 9—Impact adhesion test.

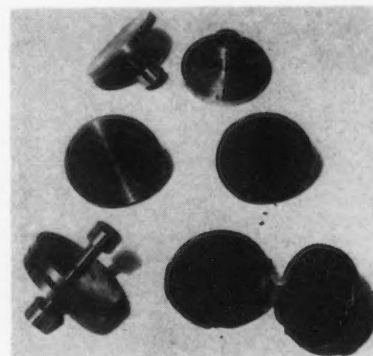


Figure 10—Impact-adhesion test plates.

necessary to fully evaluate a coating material or combination of materials. Test procedures were revised to test a larger number of samples at one time. This was particularly true for long term tests. It was also necessary to include more than one factor affecting coating performance in each test, but close control of all test conditions was maintained.

It was also recognized that the same coating properties would not be as important in the service performance of thin plastic coatings as they would be in the thick, hot-applied coating. For instance, the cold flow properties of hot-applied enamels certainly affect the resistance to soil stresses, but the hard, slick plastic films would not be subject

to this type of damage. On the other hand, the thinner coatings would be more subject to rapid deterioration by cathodic protection since many of them are brittle and subject to lifting and spalling, or could easily have a greater moisture transmission rate because of the film properties or because of application techniques. Since they are so thin, 3 to 20 mils, they must be greatly superior in most properties to materials used in thicknesses of 100 mils and upward.

#### Evaluation of Test Results

A test program such as has been described does not automatically select the best pipeline coating. It does, however, give concrete data on the various performance characteristics of materials and coating structures so that the best coating may be selected for the particular conditions encountered. Performance data and economics together govern the proper selection of a coating.

In general, long-term underground performance is of primary importance. However, construction techniques, right-

of-way, and climatic conditions are also important. End use of a coating also has a major influence in proper selection of a coating. Is the coating intended for major, long line construction, for field repairs by small crews, or in small diameter distribution system connections?

#### Summary

The best way to summarize this coating test program is to mention the benefits received from it. There are certain shortcomings to any laboratory test program; field performance is the final test for any coating. Unfortunately, a final evaluation based on field performance alone cannot be made until a coating has been in service for many years.

A test program is the means to make an evaluation of new materials for which there is no field experience to go on. Future uses can be anticipated because test coatings which under present conditions are not economically feasible may be practical in the future. Developments in protective coatings are rapid, and new materials cannot be disregarded just because present materials and techniques perform well.

Laboratory data have been collected on coatings which have proved to perform well in field experience. Testing techniques and experience to compare new, untried materials against proven ones have been gained. As stated previously, often only small differences in similar materials are found. This was true particularly when similar competitive coatings of proved performance were tested. However, unsatisfactory coatings are detected very rapidly and the reason for their failure to give good performance is determined.

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Any discussions of this article not published above  
will appear in the June, 1959 issue

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# Failure of Type 316 Stainless Steel Autoclave Components\*

By J. P. HUGO and L. G. NEL

## Introduction

THIS PAPER describes an investigation into the cause of failure of a number of autoclave components which failed in service after a surprisingly short operational life. This latter factor and, as will be seen later, the absence of a corrodant make the case in question one of special interest.

The autoclave in question was of 1 gallon capacity and non-agitated; its body and cover were of Monel while its cooling coil, thermowell, sampling tube, valve and their associated fittings were of AISI Type 316 stainless steel. The maximum operating conditions specified by the manufacturers were:

1. Maximum temperature: 343 C (650 F)
2. Maximum pressure: 2500 lb/sq inch (at 650 F)

The manufacturers considered the autoclave to be suitable for use with 50 percent maximum aqueous caustic soda (NaOH) solutions at the above-mentioned temperature and pressure maxima.

With a view to additional safety, the maximum operating conditions decided on by the operators were:

1. Temperature: 300 C (572 F)
2. Pressures at maximum temperature ranging from 1200-1500 lb/sq inch depending on the liquid charge

The temperature of the autoclave was regulated automatically by an electronic temperature controller, the controlling thermocouple being situated in the autoclave's thermowell. A comprehensive time-temperature record of every operation was obtained from a second similarly placed thermocouple connected to a recorder. After equilibrium had been established, a maximum temperature differential of  $\pm 2^\circ\text{C}$  was recorded.

## Operational History of The Autoclave

All precautions in the manufacturer's instructions were followed. Temperature was maintained at the boiling point of the liquid charge for three to four minutes with the valve open to purge the autoclave of air. Thereafter the valve was closed and the controller set at the desired operating temperature.

The operational history of the autoclave is given in Table 1. The manufacturer's specified maximum operating conditions were not exceeded or even closely approached.

Failure occurred during the night and was not detected until the following morning. Since pressure-time records were not kept, the exact instant of failure is not known. The operational life was assumed to be between 38 and 52 hours.



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## Preliminary Inspection

After failure had been detected, the autoclave was cooled and dismantled for a preliminary inspection. The nut (designated F) on the underside of the cover at the opening leading to the valve appeared to be loose. When removed, the head of the nut came loose, leaving the threaded portion in the

## Abstract

A short account is given of an investigation into the cause of failure of a number of Type 316 stainless steel autoclave components that failed in service after an extremely short operational life. Failure of these components situated in the vapor phase was attributed to stress-corrosion cracking. The active corrodant was probably chloride while the stresses that initiated failure were predominantly residual. There is evidence that the components suffered some degree of cold working prior to installation, the latter probably resulting from machining or fabrication. The failure was in no way due to the use at any time of excessive operating temperatures or pressures.

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cover. The fractured surface of nut F was completely blackened except for a few bright facets where the material had apparently still held (see Figure 1). However, no leakage had occurred at this point. The nut on the outside of the cover was intact and the valve in good order.

Further inspection showed that a similar nut (designated B) on the underside of the cover at one of the openings of the cooling coil had failed and that leakage had taken place at this point. Cracks also were observed in the cooling coil below and at a point about 2 inches from nut B.

Other nuts of the same type and the thermowell on the underside of the cover were found to be tight; no attempt was made to remove these components. Since the cover was returned to the manufacturer for dismantling and repair, it is not known whether any of the other Type 316 stainless steel components had contained cracks though no cracks were obvious on visual inspection. It should be noted that neither nuts B nor F had been touched at any time prior to failure; they had remained

TABLE 1—Operational History of Autoclave

Charge	Control Temperature, Degrees C	Maximum Pressure, lb/sq inches	Time On
	150	*	35 min
2000 cc distilled water.....	150	*	35 min
	200	205**	1 hr, 15 min
	150	60	
	200	300	
2000 cc distilled water.....	250	700	2 hr, 50 min
	300	1400	
2000 cc distilled water and two 2" x 5/16" diameter mild steel specimens.....	300	1400	25 hr, 10 min
2000 cc 10% NaOH and 2 specimens.....	300	1240-1300*** (for not less than 8 hr)	22 hr, 20 min

Total Time On: 52 hr, 35 min; Time Prior To Failure: Not Less Than 38 Hrs.

\* Denotes first two running-in periods when minor leaks were experienced at the seal.

\*\* Equilibrium condition not reached because of time factor.

\*\*\* Failure occurred.

★ Submitted for publication October 18, 1957.

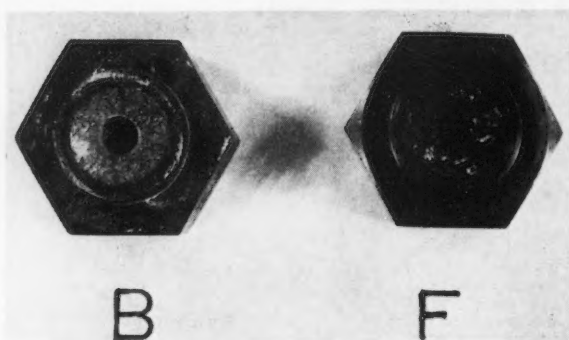


Figure 1—Top view on fracture surfaces of Type 316 stainless steel nuts B and F.

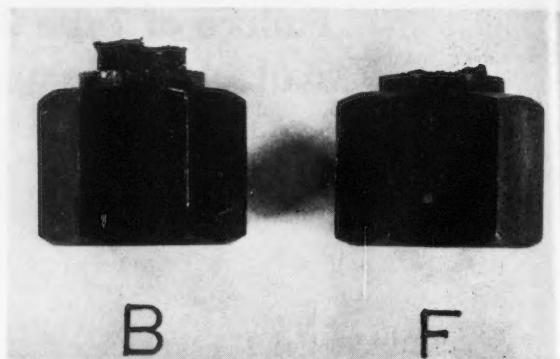


Figure 2—Side view on Type 316 stainless steel nuts B and F.

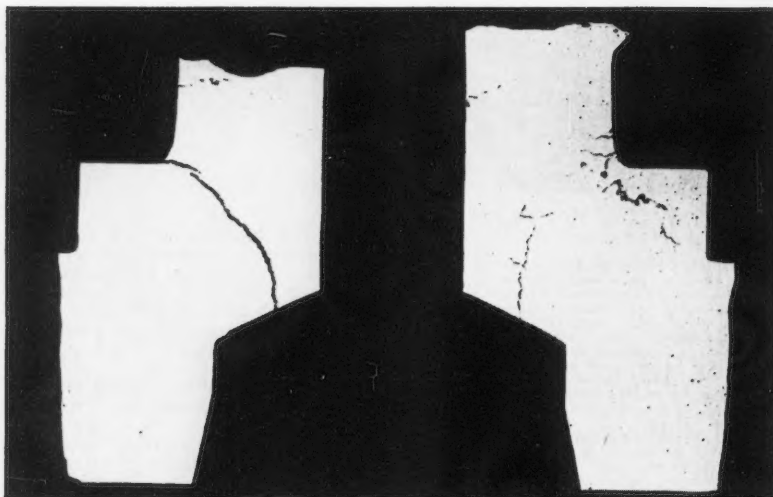


Figure 3—Longitudinal axial section, nut B, fracture horizontal at top. Unetched, 6X.

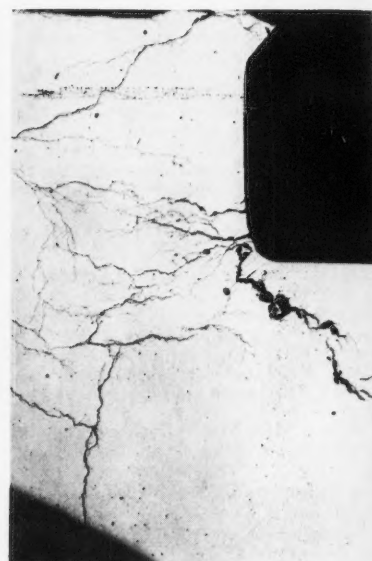


Figure 4—Longitudinal axial section, nut B, showing portion of field in Figure 3. Unetched, 12X.

in the condition and position as received from the manufacturer.

The components that failed were:

1. Nut F (AISI Type 316 stainless steel)
2. Nut B (AISI Type 316 stainless steel)
3. Cooling coil (AISI Type 316 stainless steel seamless tubing)

Plan and side views on ruptured nuts B and F are shown in Figures 1 and 2. Reference to these figures reveals that:

- (a) The fracture surface of nut F is black, denoting some corrosion
- (b) A thin white deposit is noted on nut B, which presents the possibility that leakage occurred here with consequent deposition and concentration of caustic soda at this point

(c) Nut F failed at the screw thread fillet

(d) Nut B failed at the beginning of the screw thread

Ultra-sonic, dye-penetrant and radiographic testing of the autoclave body (Monel forging) failed to reveal cracks or defects of any nature. The same applied to the cover which was tested by the manufacturer.

#### Composition of the Material of Nuts B and F

Drillings from nuts B and F were analyzed to determine if Type 316 stainless steel had been used. The results are given in Table 2.

The materials of both nuts B and F

obviously satisfy AISI specification for Type 316 stainless steel although the Mn contents are high.

#### Metallographic Examination

Both nuts B and F were sectioned through their longitudinal axes, mounted and electrolytically polished in the usual manner for a metallographic examination. A small section was also cut from the cooling coil, flattened, mounted and polished.

Figures 3 and 4 show unetched low-power views of nut B. Apart from the fracture surface, numerous branched cracks, both fine and coarse, are seen in the body of the nut. Cracks originate

TABLE 2—Chemical Analyses of Nuts B and F

Source of Sample	Composition—percentage				
	C	Cr	Ni	Mn	Mo
Nut B.....	0.06	17.49	12.75	2.48	2.5
Nut F.....	0.06	17.51	12.13	2.23	2.5

TABLE 3—Hardness of Cracked Components

Component	Hardness VPH* (Average of 5 Impressions)
Nut F, specimen MU/18.....	190
Nut F, specimen MU/19.....	199
Nut B, specimen MU/20.....	214
Cooling Coil, specimen MU/22.....	235

\*VPH—Vickers Diamond Hardness Number

from both bore and surface, the greatest concentration of cracks being situated at the screw thread fillet (see Figure 4). Nut F exhibited similar characteristics with the exception that cracking originated exclusively from the bore of the nut.

The electrolytically polished microspecimens were electrolytically etched in 3 percent sulfuric acid. In general the microstructure of nuts and cooling coil may be classified as relatively fine grained and "clean." The non-metallic inclusions were elongated, being parallel to the longitudinal axes of the nuts. This characteristic is typical of hot-rolled bar stock from which the nuts were apparently machined. The materials of nuts and cooling coil appeared to be fully austenitic because no definite evidence of ferrite was found in the microstructures. However, the materials contained a large proportion of strain markings and what was probably the quasi-martensite referred to by Edeleanu<sup>1</sup> and Hines and Hoar<sup>2</sup>. A typical example is shown in Figure 5. The distribution of the strain markings and/or quasi-martensite is uniform across any particular cross-section though there is some tendency for more markings to be situated at or near the surfaces of the components. There is no evidence that the cracks preferentially follow quasi-martensite plates.

The cracks in the nuts and cooling coil were found to be transgranular and exhibited a tendency to be branched and blunt. A typical example is shown in Figure 6. A black corrosion product was detected in the cracks.

Figure 6 shows a view on a mechanically polished and electrolytically etched microspecimen. Electrolytic polishing drastically opened-up even the fine cracks, obscuring their characteristics.

The surfaces of the components show little, if any, evidence of pitting, and the origins of the cracks do not appear to coincide with the pits.

#### Discussion

The authors are convinced that the failures in question are typical of stress-corrosion cracking which is invariably transgranular in nature in austenitic stainless steels<sup>1-7</sup>. It might be argued that transgranular cracking can also arise from fatigue or corrosion fatigue, but these phenomena are discounted as causes of failure for the following reasons:

1. The negligible number of stress cycles to which the components had been subjected
2. The virtual absence of cyclic stresses in the components which failed

The magnitude of the tensile stress required to initiate stress-corrosion cracking in austenitic stainless steels is not known with certainty (and appears to vary with the corrodant), but it is generally accepted that the threshold stress is about the same or less than the material's yield strength.<sup>2,4</sup> The latter is relatively low in fully softened 18-8 type steels. It has not been possible to place any safe limit upon the maximum allowable stress in this type alloy.<sup>8</sup>

The components under consideration are not believed to have been subjected to applied stresses of significant magnitude. In the case of the nuts (B and F), applied stresses could have two possible origins:

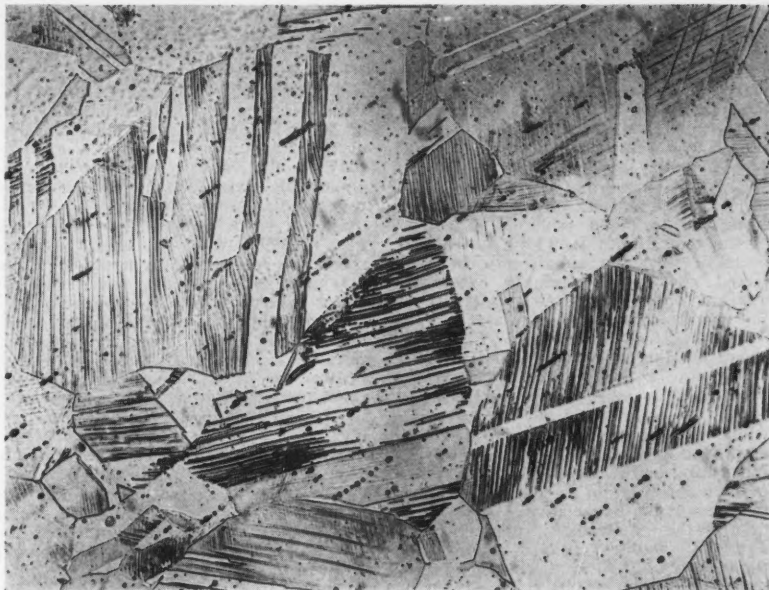


Figure 5—Micrograph showing strain markings and/or quasi-martensite, nut B. Electrolytically polished and etched, 132X.

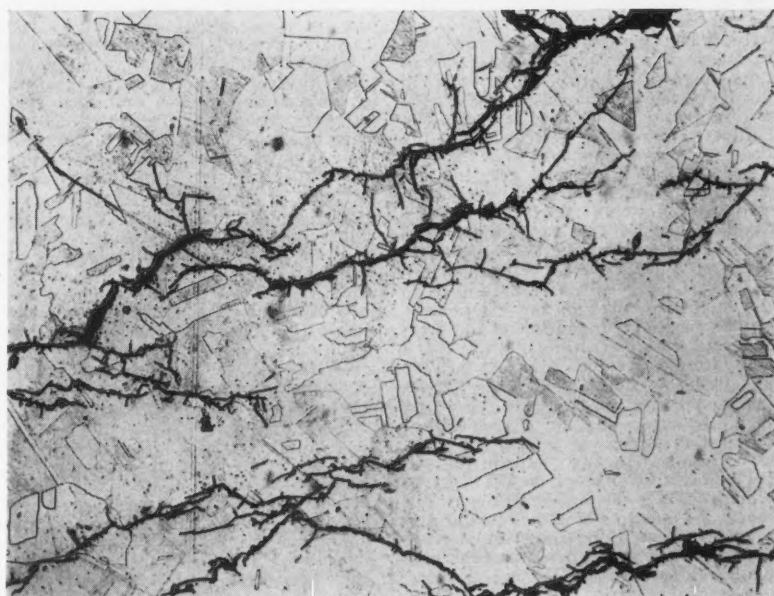


Figure 6—Micrograph showing branched and blunt transgranular cracks. Mechanically polished, electrolytically etched, 132X.

1. Stresses caused by tightening of the nuts
2. Expansion stresses caused by the dissimilar expansion coefficients of stainless steel and Monel

Applied stresses resulting from either or both of the above should not be sufficient to cause stress-corrosion cracking unless the nuts were tightened excessively, or unless insufficient design safety factors were employed. Excessive tightening of the nuts should be considered as a possible cause since the sound nuts on both the top and underside of the

autoclave cover were found to be tight. Such tightness may be necessary, however, for pressure tightness.

In the case of the cooling coil, the setting up of applied stresses of any appreciable magnitude would be difficult since this component carries virtually no load and is free to expand without constraint.

Stresses that caused failure of the components by stress-corrosion cracking must be predominantly residual in character, particularly in the case of the cooling coil. In the case of the nuts, existing residual stresses would appear



to have been reinforced by externally applied stress. Figure 4 indicates that a large number of cracks originated around nut B's screw thread fillet. A washer is situated at this fillet; thus the material at and immediately adjacent to the fillet can be expected to be subjected to a tensile stress when the nut is tightened.

Cracks also originated from the bore of the nuts and at positions removed from the threaded portions especially in the case of nut F. These latter portions of the nuts should not be subjected to any appreciable externally applied stress. Many (but not all) of the cracks appear to originate at geometrical discontinuities. This behavior is not unexpected since geometrical discontinuities act as stress raisers.

Residual stresses in austenitic stainless steels can result from the softening (quenching) heat treatments presumably employed and from deformation during fabrication and machining. The presence of such deformation or cold work should manifest itself by relatively high hardness of the material. For fully softened Type 316 stainless steel a hardness of the order of 170 Vickers Diamond Hardness would be expected. Hardness measurements in the cracked components are shown in Table 3.

From Table 3 it would appear that all the components have been cold worked to varying degrees. The cooling coil and nut B have hardnesses greater than might be expected for fully softened material though the high hardness of specimen MU/22 (cooling coil) might be partly attributable to the fact this specimen was flattened prior to mounting and testing. The hardness impressions were made in the body of the components. The surface hardnesses are probably greater, and consequently their surfaces have been cold worked to an appreciable degree. Should this be the case, these cold worked surfaces would be expected to be areas of appreciable residual stress.

The presence of quasi-martensite in the microstructures of the components provides additional evidence of the presence of considerable cold work and consequently, residual stress.

A rather puzzling aspect of the failures is found in the absence of a corrodant. Distilled water was the charge in five of the six cycles to which the autoclave was subjected; only in the last cycle was a 10 percent NaOH\* charge employed. To the authors' knowledge, Type 316 stainless steel has in the past been considered resistant to concentrated caustic soda solutions at the temperatures in question, but recent work\*

has revealed that caustic soda may, under certain conditions, cause stress-corrosion cracking in austenitic stainless steels. However, even if caustic soda were the active corrodant, that it acted with such rapidity (in less than 22 hours at 300 C) is surprising. Since cracking occurred exclusively in material exposed only to the vapor (steam) phase, the choice of caustic soda as the active corrodant is unconvincing.

Recent work by Williams and Eckel<sup>7</sup> has revealed that austenitic stainless steels are susceptible to stress-corrosion cracking in high temperature chloride water environments. This is particularly true for exposure in the steam phase of chloride bearing waters; with intermittent wetting and consequent concentration of dissolved constituents, cracking was produced with water containing as little as 1 ppm chloride. Several cases have been recorded of stress-corrosion service failures attributed to chloride ions.<sup>7, 10, 11, 12</sup>

An analysis of two typical samples of distilled water from the same source as that employed in the autoclave and a sample of the ultra-purity caustic soda showed the following results:

#### Sample Chloride Content (ppm)

Distilled water A	0.38
Distilled water B	0.24
Caustic soda	7.9

Unfortunately the oxygen content of the distilled water is not known, but this should not be high even though no deaeration precautions were employed. A 10 percent caustic soda solution was employed in the sixth and final cycle of the autoclave. The effective chloride content of this solution, due to the caustic soda, was approximately 0.79 ppm while that of the solution was about 1 ppm.

The results of Williams and Eckel<sup>7</sup> apparently justify the identification of chloride as the active corrodant in the case under consideration although the rapid failure and the low chloride content of the raw liquid charges are mystifying. It is clear, however, that no safe limit can be placed upon the allowable chloride content of the corrodant if operating conditions are such as to allow concentration of the chloride to occur.<sup>8</sup>

A mechanism of concentration of chloride in parts of the autoclave exposed to the steam phase may be found in such factors as water carry-over, splashing, and start and shut-down operations. In the case under consideration these factors can be discounted since splashing and water carry-over could have occurred only to a limited extent in the autoclave. It was completely sealed, not agitated and not con-

trolled by the use of a controlled leak or blow-off device. Only six start and five shut-down operations were carried out prior to failure. Nevertheless, all the evidence available would appear to point to some chloride concentration mechanism having been operative.

#### Conclusions

The failure in service of a number of Type 316 stainless steel autoclave components, situated in the vapor (steam) phase, is attributed to stress-corrosion cracking. The active corrodant is probably chloride while the stresses that initiated failure are predominantly residual. There is evidence that the components had suffered some cold working prior to installation, the latter probably resulting from machining or fabrication. The failure is in no way due to the use at any time of excessive operating temperatures or pressures.

#### Acknowledgment

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\*The NaOH employed was of ultra pure quality. (Mercks Reagenzien 6496)

Any discussions of this article not published above  
will appear in the June, 1959 issue

# Corrosion Properties of Tantalum, Columbium, Molybdenum and Tungsten\*

By CLIFFORD A. HAMPEL

## Introduction

**T**ANTALUM, COLUMBIUM, tungsten and molybdenum are characterized by their high melting points, their high strength at elevated temperatures and their unique corrosion resistant properties. The object of this paper is to summarize these properties and to indicate the present and potential areas of application in the chemical industry.

In Table 1 some of the physical properties of these metals are presented. Also included are the density and melting points of a low carbon steel by way of comparison.

As to general chemical properties, the four metals are transition elements, columbium and tantalum being located in Group V-A in the periodic table below vanadium, and molybdenum and tungsten in Group VI-A below chromium. All are multivalent elements capable of existing as cations and in anionic radicals. They all form borides, carbides, nitrides, oxides, halides, and sulfides. The oxide film naturally present on the surface of items fabricated from these metals has a great deal to do with their corrosion resistant properties. This is especially true for tantalum.

As is to be expected, the specific chemical reactivity, and in turn the corrosion resistance of each metal varies with the metal and with the temperature and concentration of the reagent.

## Tantalum

Tantalum, the most resistant of the four metals, is inert to acidic solutions, with the exception of those involving HF and free  $\text{SO}_3$ , at the temperatures normally encountered in industrial processes. In the case of 98 percent  $\text{H}_2\text{SO}_4$  and 85 percent  $\text{H}_3\text{PO}_4$ , a slow uniform attack begins at about 175 C. Figure 1 shows the curves relating temperature and the rate of attack in inches per year for fuming (15 percent  $\text{SO}_3$ )  $\text{H}_2\text{SO}_4$ , 98 percent  $\text{H}_2\text{SO}_4$  and 85 percent  $\text{H}_3\text{PO}_4$ . The first curve confirms the rapid attack due to free  $\text{SO}_3$ , and the others the low rates of corrosion below 200 C for 98 percent  $\text{H}_2\text{SO}_4$  and 85 percent  $\text{H}_3\text{PO}_4$ . As the concentration of  $\text{H}_2\text{SO}_4$  is decreased the rate of corrosion at a given temperature becomes less. For example, a recent test by a chemical company showed a rate of 0.0031 inch per year for 90 percent  $\text{H}_2\text{SO}_4$  at 250 C. This is about one-tenth that for 98 percent  $\text{H}_2\text{SO}_4$  at the same temperature.

Tantalum is inert to a wide variety of reagents over the temperature range commonly used in solution processes.

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## About the Author



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Among them are nitric acid, including red fuming  $\text{HNO}_3$ , hydrochloric acid, aqua regia, chromic acid, perchloric acid, nitrogen oxides, chlorine oxides, chlorine, bromine, hypochlorous acid, organic acids including monochloroacetic, and hydrogen peroxide, all with no concentration limitations.

## Exposure in Nitric Acid

To illustrate the wide range of temperatures and concentrations in which tantalum is effective, data recently reported on exposures in nitric acid are of interest. Montecatini, the huge Italian chemical company in Milan, exposed tantalum to 98 percent  $\text{HNO}_3$  containing 0.1 percent  $\text{H}_2\text{SO}_4$ , 0.4 percent nitrogen oxides and 1.5 percent  $\text{H}_2\text{O}$  at 132 C for 762 hours. The corrosion rate was 0.00002 inch per year and the metal was unchanged in appearance.

In another test in the laboratory of an American company, tantalum was

## Abstract

The corrosion resistance properties of tantalum, columbium (niobium), molybdenum and tungsten as they are affected by the nature of the corrosive medium, its concentration and its temperature are presented. Tantalum, the most resistant, is inert to all acids except HF and free  $\text{SO}_3$  at temperatures usually encountered in industrial processes. It is attacked by strong alkalis at room temperature. The resistance to gases depends upon the temperature of exposure. Columbium in general is less resistant than tantalum in all media. Molybdenum shows good resistance to non-oxidizing acids and to alkaline solutions over a wide range of temperatures and concentrations, and is attacked only slightly by hydrofluoric acid. Tungsten's resistance to acid corrosion is equal to or better than that of molybdenum. All four are extremely resistant to the liquid metals at elevated temperatures. The structural and fabrication limitations of each metal are discussed and the useful fields of application are indicated. 63.1

exposed to 70 percent  $\text{HNO}_3$  containing 0.49 moles/liter of aromatic compounds at 180-200 C and 200-250 psi. The solution was aerated. A corrosion rate of 0.0004 inch per year was calculated, but since the test was only of 14 hours duration and the sample weight loss 0.0004 gram, the corrosion rate may be considered nil.

A tantalum lined autoclave has been operated for several months with a 25 percent  $\text{HNO}_3$  solution at 250-300 C and 1200-1500 psi in a chemical company's pilot plant. While the tantalum has become colored by an oxidation film, the corrosion of the metal has been very low, an estimated 1 to 3 percent of the original metal.

## Effect of Fluoride Ion

Hydrofluoric acid, gaseous HF, or acid solutions containing more than 2 or 3 parts per million of fluoride ion all corrode and embrittle tantalum. However, this attack apparently does not occur when fluoride ion is present in chromium plating baths. In one test a corrosion rate of 0.00002 inch per year was observed on a tantalum coupon hung in a chromium plating bath for 2½ months. The 40 percent  $\text{CrO}_3$  contained 0.5 percent fluoride ion and was maintained at 130-140 F. In another test

TABLE 1—Some Physical Properties of Ta, Cb, W and Mo Compared with 1030 Steel

METAL	Atomic Number	Atomic Weight	Density, g/cc	MELTING POINT,	
				Degrees C	Degrees F
Tantalum.....	73	180.88	16.6	3000	5432
Columbium.....	41	92.91	8.57	2415	4380
Tungsten.....	74	183.92	19.3	3400	6152
Molybdenum.....	42	95.95	10.2	2625	4760
Steel (1030).....	..	.....	7.86	1525	2777

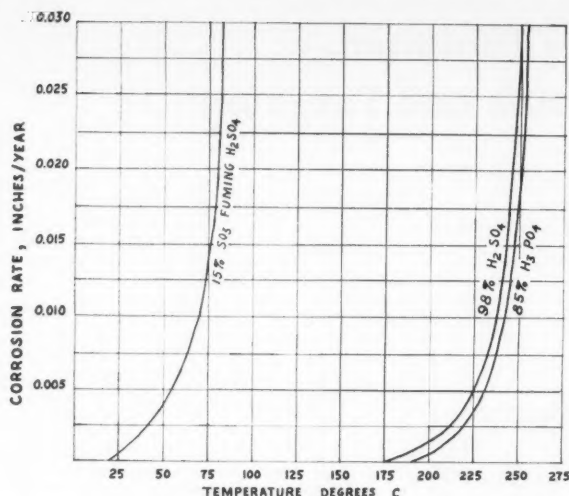


Figure 1—Corrosion rate versus temperature of tantalum in 15 percent  $\text{SO}_3$  fuming  $\text{H}_2\text{SO}_4$ , 98 percent  $\text{H}_2\text{SO}_4$ , and 85 percent  $\text{H}_3\text{PO}_4$ .

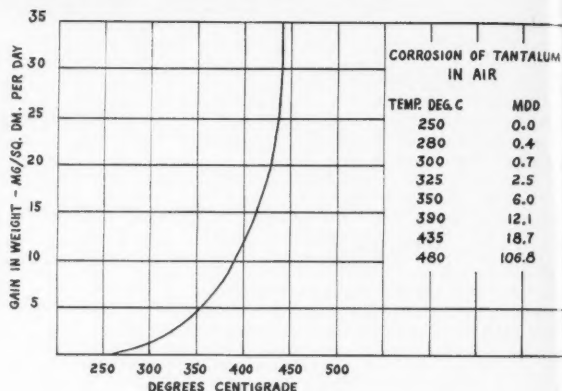


Figure 2—Corrosion of tantalum in air, expressed as weight gain in mg/sq dm/day, versus temperature.

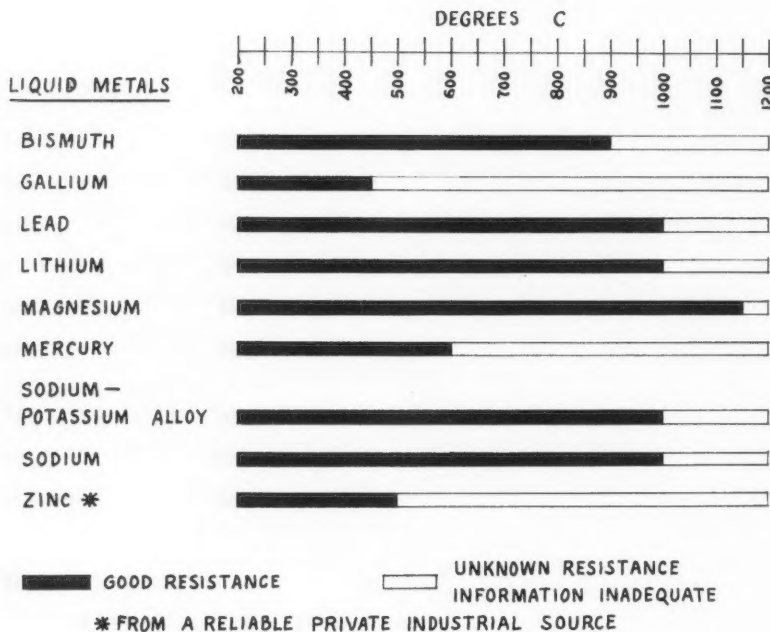


Figure 3—Corrosion resistance of tantalum to liquid metals as affected by temperature.

of 96 hours duration a corrosion rate of 0.0002 inch per year was measured. This bath contained 33 oz/gal  $\text{CrO}_3$ , 0.20 oz/gal  $\text{H}_2\text{SO}_4$  and 0.40 oz/gal fluoride ion and was held at 130 F. The negligible attack on tantalum by the fluoride ion in these strongly acidic solutions may be an inhibition caused by complex ion formation between fluoride and chromium ions.

Tantalum is attacked even at room temperature by concentrated alkaline solutions and is dissolved by molten alkalis and molten sodium pyrosulfate. However, tantalum is fairly resistant to dilute alkaline solutions. In one long

term exposure test in a paper mill it suffered no change in a solution of pH 10. It has been used as anode baskets in a number of silver cyanide barrel platers for several years, and although the solutions are quite alkaline with free KOH, the tantalum has remained bright and ductile with no failures occurring.

Tantalum readily absorbs atomic hydrogen even at room temperature and molecular hydrogen at temperatures above about 250 C, forming an interstitial combination whose maximum hydrogen ratio is  $\text{TaH}_{0.74}$ . The metal becomes very hard and brittle. Most frequently, the absorption of atomic hy-

drogen occurs when the tantalum becomes cathodic in galvanic cell circuit. Under these conditions hydrogen is discharged on the tantalum cathode. Stray voltages give the same result. If the tantalum is maintained anodic, as by impressing 2 to 10 volts upon it, or if it is electrically insulated, the formation and absorption of hydrogen is prevented.

The oxide film on the surface of tantalum prevents the flow of current from the tantalum to an electrolyte when the metal is made anodic, although it permits the passage of current when a metal to metal contact occurs. This property makes tantalum valuable for rectifier and capacitor applications.

At temperatures higher than those associated with solution processes, tantalum becomes a reactive element. While it is completely stable in air at 250 C and below, at 300 C it shows a tarnish in 24 hours and the rate of corrosion (weight increase) increases rapidly at higher temperatures. This is shown in Figure 2 which is a plot of the weight gain in mg/dm<sup>2</sup>/day versus temperature. Similar effects are obtained in oxygen and in nitrogen.

Fluorine attacks tantalum at room temperature, chlorine at about 250 C, bromine at 300 C and iodine at well above 1000 C. Sulfur reacts with tantalum at red heat forming tantalum sulfide,  $\text{Ta}_2\text{S}_5$ .

#### High Temperature Exposure

Tantalum is resistant to reaction with most of the liquid metals at high temperature. It is not affected by sodium, potassium, sodium-potassium alloys (NaK), lithium and lead at 1000 C, by bismuth at 900 C, by mercury at 600 C, by zinc at 500 C, and by gallium at 450 C. Recent work at Argonne Laboratories shows it to be unattacked by magnesium, and uranium-magnesium and plutonium-magnesium alloys at 1150 C.<sup>1</sup> Figure 3 is a bar chart of these data. This resistance makes tantalum of great interest as a material of construction for nuclear energy power systems where



liquid metals are used as heat transfer agents.

Aluminum reacts rapidly in the liquid state with tantalum to form the stable compound,  $Al_3Ta$ , aluminum tantalide.

### Columbium

While somewhat similar to tantalum in corrosion resistance, columbium is attacked slowly by most of the reagents to which tantalum is inert and rapidly by others. In no instance is it unaffected by materials which corrode tantalum.

Columbium has a high degree of resistance to most acids and acidic solutions at room temperature, one exception being 10 percent oxalic acid. Hot concentrated hydrochloric, sulfuric and phosphoric acids attack the metal, although hot concentrated nitric acid does not. It is unaffected at room temperature by sulfuric, hydrochloric, nitric, phosphoric, tartaric, lactic, acetic and perchloric acids, aqua regia, 5 percent phenol, ammonium hydroxide, 30 percent hydrogen peroxide and 10 percent ferric chloride.

Like tantalum, columbium absorbs and becomes embrittled by atomic (nascent) hydrogen. Thus, those acids which attack it even slowly liberate sufficient hydrogen to cause the metal to become brittle. For example, while the corrosion rate in 10 percent oxalic acid at 21 C was found to be only 0.0006 inch per year, the sample of columbium became brittle in 82 days.<sup>2</sup> Precautions must be taken to prevent the metal from becoming cathodic in a galvanic couple in electrolytes since the hydrogen discharged upon it causes embrittlement.

Columbium is not resistant to alkaline solutions (i.e., 5 percent NaOH or KOH and 20 percent  $Na_2CO_3$ ), although it is not attacked by ammonium hydroxide at room temperature.

Air begins to oxidize columbium slowly at about 230 C, the rate accelerating as the temperature is increased. At 390 C a white oxide begins to appear on the surface. Heating in pure nitrogen results in nitride formation above 300 C. Columbium reacts rapidly with fluorine at room temperature, with chlorine at about 200 C and with bromine at somewhat higher temperatures.

The resistance of columbium to liquid metals at elevated temperatures approximates that of tantalum. This resistance plus the low thermal neutron absorption cross section of columbium makes its use attractive in some types of nuclear reactors, chiefly as a canning material for uranium.

### Molybdenum

While molybdenum has very attractive corrosion resistance properties, the great difficulty of obtaining strong, non-brittle welds with it has restricted its application in chemical equipment.

The metal is outstanding for its resistance to cold or hot hydrofluoric acid and to cold or hot concentrated hydrochloric acid, provided oxidizing agents are not present. Concentrated nitric acid attacks molybdenum slowly at room temperature probably because of the formation of a passivating film, but dilute nitric acid and hot concentrated nitric acid react with it rapidly, as does a mixture of HF and  $HNO_3$  at 20 C. It is dissolved readily by aqua regia.

The corrosion of molybdenum by sulfuric acid varies with the temperature and concentration. The presence of air or oxygen in the sulfuric acid system accelerates the corrosion rate. While the metal reacts readily with boiling concentrated  $H_2SO_4$ , the corrosion rate in boiling (305 F) 65 percent  $H_2SO_4$  is about 0.001 inch per year as determined by a test in an industrial sulfuric acid concentrator.

When used as an anode in 20 percent sulfuric acid the corrosion rate at room temperature is 612 mg per ampere-hour, 8.3 inches per year, at a current density of 370 amperes per square foot.<sup>3</sup> This study was made to find suitable support members for electrocleaning operations in sulfuric acid. Tantalum could be used with immunity from corrosion in this application.

The attack by phosphoric, acetic and chromic acids is slight.

Molybdenum is not corroded by alkaline solutions. However, when made an anode in caustic solutions it dissolves

rapidly. Molten sodium or potassium hydroxide attack molybdenum beginning at about 660 C in the absence of oxygen. In the presence of oxygen the attack begins at a lower temperature.

Fluorine attacks molybdenum at 20 C, reaction with chlorine begins at about 250 C, and with bromine at about 450 C. The metal is unaffected by iodine at 800 C.

Oxygen and air react with molybdenum at elevated temperatures. Oxidation begins at 300 C and at somewhat above 600 C the oxide,  $MoO_3$ , begins to sublime from the surface. While molybdenum has excellent strength at high temperatures, it must be protected from the air due to this poor oxidation resistance. A coating of molybdenum disilicide is one of the more successful methods of preventing oxidation.

In nitrogen, nitriding begins at 1500 C; in carbon monoxide, carburizing starts at 1400 C, and in carbon dioxide, oxidation begins at 1200 C. Steam attacks molybdenum at 700 C. The metal

TABLE 2—Degree of Attack on Metals by Sulfuric Acid

METAL	DILUTE, 20-25%		CONCENTRATED, 98%	
	20 C	100 C	20 C	100 C
Tantalum.....	None	None	None	None <sup>1</sup>
Columbium.....	None	None	0.00009 ipy <sup>2</sup>	0.019 ipy <sup>3</sup>
Molybdenum.....	Very slight	Slow	Slow	Slow <sup>4</sup>
Tungsten.....	None	Slow	Slow	Slow

<sup>1</sup> Corrosion rate, 175 C, 0.0001 ipy; 200 C, 0.0015 ipy.

<sup>2</sup> Partially embrittled in one year.

<sup>3</sup> Brittle.

<sup>4</sup> Corrosion rate, 65 percent  $H_2SO_4$ , boiling point (152 C), 0.001 ipy.

TABLE 3—Degree of Attack on Metals by Hydrofluoric Acid

METAL	DILUTE		CONCENTRATED	
	20 C	100 C	20 C	100 C
Tantalum.....	Rapid	Rapid	Rapid	Rapid
Columbium.....	Rapid	Rapid	Rapid	Rapid
Molybdenum.....	Slight	Slight	Slight	Slight
Tungsten.....	Slight	Slight	Slight	Slight

TABLE 4—Degree of Attack on Metals by Sodium Hydroxide

METAL	DILUTE		CONCENTRATED	
	20 C	100 C	20 C	100 C
Tantalum.....	Slow	Slow	Rapid	Rapid
Columbium.....	0.001 ipy	0.018 ipy <sup>1</sup>	Rapid	Rapid
Molybdenum.....	None	None	None	None
Tungsten.....	None <sup>2</sup>	None <sup>2</sup>	Slight <sup>2</sup>	Slight <sup>2</sup>

<sup>1</sup> Brittle.

<sup>2</sup> In the absence of oxygen.

TABLE 5—Degree of Attack on Metals by Hydrochloric Acid

METAL	DILUTE		CONCENTRATED	
	20 C	100 C	20 C	100 C
Tantalum.....	None	None	None	None
Columbium.....	0.00004 ipy	.....	0.0001 ipy	.004 ipy <sup>1</sup>
Molybdenum.....	Very slight	Slow	Very slight	Slight
Tungsten.....	None	Slight	None	Slight

<sup>1</sup> Brittle.

TABLE 6—Degree of Attack on Metals by Nitric Acid

METAL	DILUTE		CONCENTRATED	
	20 C	100 C	20 C	100 C
Tantalum.....	None	None	None	None
Columbium.....	None	None	None	None
Molybdenum.....	Rapid	Rapid	Slight*	Rapid
Tungsten.....	Slight	Slight	Slight	Slight

\* Forms protective film.

does not react with hydrogen and sintering of bars of compacted powder is conducted at temperatures up to 2200 C in hydrogen.

Sulfur reacts with molybdenum at red heat, and hydrogen sulfide at 1200 C.

The resistance of molybdenum to liquid metals at elevated temperatures is outstanding, comparable to that of tantalum. Molybdenum is reported<sup>4</sup> to be suitable for long-time service with sodium up to 1500 C, with bismuth to 1427 C, with lead to 1100 C, with lithium, potassium and sodium-potassium alloys to 900 C, with bismuth-lead alloys to 800 C, with magnesium to 610 C, with mercury and bismuth-tin-lead alloys to 600 C, and with gallium to 300 C. It is severely attacked by tin at 1000 C, and aluminum attacks it fairly rapidly even at the melting point of aluminum, 660 C.

#### Tungsten

Like molybdenum, tungsten exhibits very interesting corrosion resistance properties. However, it is most difficult to weld and is brittle at room temperature, except in thin sections. For these reasons it has not been used as a material of construction for chemical equipment. The chief applications of tungsten are found in electronic tube components, x-ray targets, electrical contacts and welding electrodes where its high melting point, low vapor pressure, and high strength are attractive factors.

Tungsten is not attacked by hydrofluoric acid and only slightly by hot dilute or concentrated hydrochloric acid and hot concentrated sulfuric acid. The attack by nitric acid and by aqua regia is slow, but a mixture of HF and HNO<sub>3</sub> dissolves tungsten rapidly.

In the absence of oxygen, tungsten resists corrosion by alkali solutions and ammonium hydroxide. Molten caustic alkalis in the presence of oxygen react rapidly with tungsten. When used as an anode in alkaline solutions, the metal dissolves rapidly. When used as an anode at room temperature in 20 percent sulfuric acid, tungsten corrodes at the rate of 0.0013 inch per year.<sup>5</sup> This low corrosion rate has been found to apply in commercial electrolytic operations involving dilute sulfuric acid where tungsten has been used as an anodic contact.

Fluorine attacks tungsten at room temperature, chlorine at about 250 C and bromine and iodine at red heat. In the presence of oxygen, chlorine attacks tungsten at 600 C.<sup>5</sup>

Water vapor reacts rapidly with tungsten at 700 C. Oxidation of the metal in air begins at about 400 C, and the rate becomes rapid at above 600 C.

Tungsten is very stable in nitrogen and nitriding begins at 2300 C. Hydrogen has no effect at any temperature. In carbon dioxide, oxidizing of tungsten begins at 1200 C, and in carbon monoxide, carburizing begins at 1400 C. Hydrogen sulfide discolors tungsten at 1200 C.

Elements such as carbon, boron, silicon, and sulfur combine with tungsten at elevated temperatures to form corresponding compounds. For example, tungsten carbide, WC, begins to be formed at 1200 C when carbon and tungsten are in contact.

Tungsten has good resistance for extended use in many liquid metals. It is suitable for use with sodium and sodium-potassium alloys to 900 C, with mercury to 600 C, with gallium to 800 C, with bismuth to 980 C, and with magnesium to 600 C. It is reported to have disintegrated at 1000 C in lithium contained in Armco iron.<sup>6</sup>

#### Comparative Corrosion Effects

The effect of each of several common corrosive agents upon the four metals is presented in Tables 2, 3, 4, 5, and 6.

#### References

1. I. O. Winsch and L. Burris, Jr. *Chem. Eng. Progress*, 53, No. 5, 237-42 (1957).
2. C. W. Balke, *Corrosion Handbook* (H. H. Uhlig, Editor), New York, John Wiley & Sons, 1948, p. 60.
3. F. R. Morral and J. L. Bray, *Trans. Electrochem. Soc.*, 75, 427-440 (1939).
4. Arc-Cast Molybdenum and its Alloys. Climax Molybdenum Co. New York, 1955, p. 39.
5. R. Spitzin and L. Kashtanow, *Z. anorg. Chem.*, 157, 154 (1926).
6. The Reactor Handbook, Vol. 3, Section 1: "General Properties of Materials", AECD-3647, U. S. Atomic Energy Commission, 1955.

#### DISCUSSION

**Question by Otto H. Fenner, Monsanto Chemical Company, St. Louis, Missouri:**

It has been stated that tantalum plugs used in patching glass lined steel equipment containing plugs of other alloys such as Hastelloy C suffer galvanic corrosion in an accelerated manner by completion of circuit through the electrolyte and steel shell. Will you please comment on the authenticity and, if true, mechanism of failures of tantalum by this means?

**Reply by Clifford A. Hampel:**

Tantalum is cathodic to many metals, including high nickel alloys like Hastelloy C, and when it becomes cathodic in a galvanic circuit, like the one referred to, atomic hydrogen is discharged upon its surface. The hydrogen so formed is absorbed and the tantalum becomes em-

brittled and frequently decrepitates, since tantalum hydride is a brittle and friable material. Tantalum should not be used to repair a glassed steel vessel which contains repair plugs or patches of another metal, nor should repair plugs of another metal be inserted into a wall of a vessel containing tantalum repair plugs. If the formation of an electrolytic circuit is avoided or prevented, no danger to the tantalum exists.

**Question by William L. Wilson, Columbia Southern Chemical Corp., Barberton, Ohio:**

What are the relative costs of tantalum, columbium, molybdenum and tungsten at the present time?

**Reply by Clifford A. Hampel:**

At the present time the costs of 0.020 inch sheet made by powder metallurgy techniques are \$50.00 per pound for tantalum, \$65.30 for columbium, \$19.90 for molybdenum and \$33.00 for tungsten. Prices for other forms do not necessarily follow the same ratios since the above prices include the cost of converting powder or ingots to the sheet form.

**Question by Eugene H. Edwards, Orinda, Calif:**

What is the mechanism by which tantalum is embrittled by hydrogen?

**Reply by Clifford A. Hampel:**

Probably the best way of discussing the mechanism by which tantalum is embrittled by hydrogen is to consider what happens when hydrogen is absorbed by tantalum. While the state of the hydrogen in the metal is not known, it probably is not present in true chemical combination, but in solid solution or as an interstitial compound. Further, the hydrogen is present as atoms or ions, not as molecules. The metal lattice is expanded by the absorption of the hydrogen, thereby stressing the lattice so that the metal becomes brittle. This is accompanied by a definite decrease in density and by an increase in the molecular volume of the tantalum. The absorbed hydrogen is liberated when the hydrogen-containing metal is heated to 1200 C and cooled in a vacuum.

It might be pointed out that the embrittling mechanism is no different from that which occurs in the hydrogen absorption by other metals in this same class, such as zirconium, titanium, columbium and uranium, all of which undergo similar embrittlement caused by hydrogen.

Any discussions of this article not published above will appear in the June, 1959 issue

# NACE TECHNICAL COMMITTEE REPORT

## Criteria for Adequate Cathodic Protection Of Coated,\* Buried or Submerged Steel Pipe Lines and Similar Steel Structures

A Report of NACE Technical Unit Committee T-2C  
On Criteria for Cathodic Protection\*\*

### Abstract

Criteria are given for the adequate cathodic protection of coated, buried or submerged steel pipe lines and similar structures. It was recommended that a protective electric current be applied to the structure in an amount sufficient to maintain its external surface, at every point, negative by at least 0.85 volt to a copper-saturated copper sulfate half cell in the structure's immediate proximity. Three reservations regarding the use of these criteria are incorporated into the recommendations. 5.2.4

**B**ASICALLY, PROTECTIVE electric current must be applied to the structure being protected in an amount sufficient to prevent current flow into the soil from any and all points on the surface of the structure.

Specifically, this protective electric current must be applied to the structure in an amount sufficient to maintain its external surface, at every point, negative by at least 0.85 volt to a copper-saturated copper sulfate half cell in the immediate proximity thereof.

The following reservations should be noted:

- (1) Any departure from the above specified *minimum value* of 0.85 volt will be acceptable only if such departure is shown to be within sound engineering principles, and necessary to meet an abnormal condition.
- (2) It is recognized that a high degree of engineering judgment is required to

\* Coatings should meet requirements specified in the statement of minimum requirements for protection of buried pipe lines prepared by a Task Group of NACE Group Committee T-2. This report was published in *Corrosion*, 12, 479t (1956) Oct.

\*\* Ray M. Wainwright, Good-All Electric Manufacturing Company, Ogallala, Nebraska, chairman.

conduct a potential survey, necessarily limited to the determination of potentials at a finite number of points, and then to conclude therefrom that the minimum potential has been achieved at every point.

- (3) It is recognized that in some circumstances it may be economically impractical to maintain a given structure under cathodic protection; the presence of other structures in the vicinity may result in interference in either direction. Under such circumstances, plans for the application of adequate cathodic protection should be submitted to the local corrosion coordinating committee for coordination. Where no such committee exists, it is recommended that consultations be held with the owners of structures which may be involved.



### TECHNICAL PAPERS ON CORROSION WELCOMED

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.



# Kinetics of Hydrogen Absorption, Desorption And Permeation of Metals\*

By T. G. OWE BERG

## Introduction

THE SUBJECT of this paper is the mechanisms by which hydrogen is absorbed by and desorbed from metals. The effects of hydrogen will be commented upon briefly because they are frequently not distinguishable from the mechanisms under consideration.

Hydrogen may be held in a metal in several ways:

1. As interstitially held H atoms.
2. As H atoms held on lattice sites, i.e. as a hydride.
3. In compounds in voids or at grain boundaries.

This paper is concerned only with interstitially held H atoms in structural materials such as Fe, Cu, Al, and their alloys. The amounts of hydrogen held interstitially are comparatively small, a few parts per million by weight.

The hydrides of these metals are not stable at, or even below, room temperature. Yet, the hydride will be assumed to form as an intermediate product in the mechanism of absorption. The implications of this assumption will be discussed later.

## Retained Hydrogen

Heavily charged with hydrogen, steel develops blisters caused by high pressure hydrogen gas in voids. The pressure in such blisters is much higher than atmospheric pressure, otherwise it would not suffice to deform the steel. This indicates that H<sub>2</sub> molecules do not pass through steel and that H<sub>2</sub> gas decomposes very slowly into H atoms, which do pass through the steel. However, in vacuum fusion analysis for hydrogen, all hydrogen, whether dissolved or included, is determined.

In the presence of elements which form molecular compounds with hydrogen, such compounds may also be formed in voids and possibly at grain boundaries. These pass through the steel no more than H<sub>2</sub> molecules. Their decomposition may be more or less sluggish, but the passage through the steel of H atoms held in such compounds is certainly delayed by the decomposition process. This phenomenon has been observed by a number of investigators in connection with analysis for hydrogen. Reference may be made to Portevin, Chaudron and Moreau,<sup>1</sup> who observed that the amount of H<sub>2</sub> evolved from Fe at 300 C was essentially smaller than that evolved at 600 C. The difference was attributed to a difference between H held in the lattice and H held at grain boundaries. Kelemen and Yang<sup>2</sup> studied the evolution of H<sub>2</sub> from electrodeposited Ni. They found that part of the H came out rapidly at room temperature or slightly higher temperatures while the rest came out slowly unless the tempera-

\* Submitted for publication October 18, 1957.



About  
the  
Author

T. G. OWE BERG is employed by Douglas Aircraft Company, Inc., Long Beach, Cal. His principal field of work is in reaction kinetics, surface reactions and physical chemistry. He graduated in 1939 from the Royal Institute of Technology School of Technical Physics, Stockholm. He received a doctorate degree in 1954.

ture was raised above 550 C. These investigators also distinguish between dissolved and retained hydrogen. Andrew et al.,<sup>3</sup> and Bach, Dawson and Smith<sup>4</sup> studied the H<sub>2</sub> evolution from steel and cast iron, respectively, at various temperatures. Their results show a complex relation of rate of H<sub>2</sub> evolution and level of hydrogen solubility to temperature.

The phenomenon of hydrogen retention in molecular compounds is an essential feature of the behavior of hydrogen in metals. From the viewpoint of absorption and desorption of interstitially held H atoms, as a source of hydrogen a compound in a void inside the metal is not different from the same compound outside the metal.

It is frequently held that migration of H atoms in steel is a slow process. This view is based on experiences in which retained hydrogen must have been involved. Thus, the rate of migration of H atoms is equal in a large casting and in a small casting, but the probability of the formation of compounds on the passage through a large casting must be greater than in the case of a small casting, disregarding possible differences in amount and distribution of compound-forming elements in the steel.

There is evidence to the effect that hydrogen as such does not cause embrittlement and other hydrogen defects but that these phenomena require that retained hydrogen be formed.

## Dissolved Hydrogen

Experience indicates that steel does not absorb hydrogen from dry hydrogen gas. Thus, hydrogen gas may be stored in steel containers. The mechanism of hydrogen absorption from hydrogen gas contains the formation of H atoms as

## Abstract

The reaction mechanisms for the absorption of hydrogen by metals from moist hydrogen gas and dilute acid solutions, for the desorption from metals of dissolved hydrogen in the presence of water and for hydrogen solubility and permeation under those conditions, are discussed. Their kinetics are also given. Formulae are derived for rates and equilibria. These are compared with experimental data. The migration of H atoms in the metal is concluded to be associated with zero or nearly zero activation energy. Diffusion is rapid enough to maintain almost uniform distribution of H atoms in the metal during absorption and desorption under ordinary conditions.

3.8.4

a slow process. In the absorption of hydrogen from an acid solution, H atoms are formed as a result of reactions between metal and solution. The rate of formation of H atoms is related to the rate of reaction and to the rate of dissolution of the metal. Absorption of hydrogen usually requires the supply of H atoms to the metal. The formation of H atoms from the source of hydrogen is an important part of the absorption mechanism.

Hydrogen absorbed by steel in processing may be given off in storage. Desorption of hydrogen occurs also during absorption. When the rates of absorption and desorption are equal, the hydrogen in the steel is at equilibrium with the source of hydrogen. The concentration of H in the steel is then the solubility of hydrogen in the steel under those experimental conditions.

The solubility is not merely a property of the metal but depends upon the environment to which it is exposed.

At equilibrium, the dissolved hydrogen may be assumed to be distributed uniformly in the metal. During absorption or desorption in a state widely off equilibrium, the distribution is determined by the rate of migration of H atoms in the metal as compared to the rate of absorption or desorption at the surface. The concentration of H close to the surface is affected immediately by absorption and desorption while that in the interior is affected indirectly by those processes through migration of H atoms.

The rates at which H atoms enter and leave the surface are determined by surface processes. The rates depend on the concentration of H at the surface and on the environment with which H atoms are exchanged. For a given set of experimental conditions, the boundary condition is

$$\frac{\partial c}{\partial t} = f(c) \quad (1)$$

when  $f(c)$  is the net rate of absorption, i.e. the difference between the rates of absorption and desorption. The distri-

bution of H atoms inside the metal may be expressed by the diffusion equation

$$\frac{\partial c}{\partial t} = D \Delta c \quad (2)$$

subject to the boundary condition (1),  $\Delta$  denoting Laplace's operator.

When the surface concentration increases gradually from zero,  $f(c)$  decreases, passes through zero for a certain value of  $c$ , and then decreases further. The diffusion constant  $D$  is independent of the concentration, at least for the low concentrations under consideration in this paper. The rate of diffusion is proportional to the concentration gradient. Therefore, in principle, there must be a concentration gradient, however small, in order that the absorbed H be distributed over the metal and the surface concentration be kept low enough to allow absorption to proceed or high enough to allow desorption to proceed. The concentration gradient need be no greater than that the surface concentration be kept at such a level. Therefore, the smaller the rate of the surface processes, the smaller the concentration gradient and the more uniform the distribution in the metal.

From the viewpoint of practical applications, the surface processes are of primary interest since these may be controlled by various means, whereas the diffusion constant is beyond control. Particularly interesting are the rates of the surface processes in relation to the magnitude of the diffusion constant. The actual distribution in the metal is important only in relation to the extent it deviates from the uniform distribution. The distribution during the approach to equilibrium should, of course, be distinguished from the distribution owing to compound formation which may vary in a large casting with the distribution of compound-forming elements established in solidification.

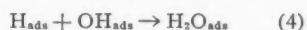
#### Absorption and Desorption Mechanisms

The heat of dissociation of the  $H_2$  molecule is 102 kcal/mol. At technically attainable temperatures, only a small fraction of the  $H_2$  molecules possess that energy, e.g.  $e^{-20}$  at 2000 K. The homogeneous formation of H atoms in pure  $H_2$  gas is therefore a slow process. The heterogeneous decomposition of  $H_2$  in the absence of catalysts is no more rapid. Furthermore, the recombination of H atoms is a slow process in the absence of catalysts.

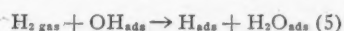
Moist  $H_2$  reacts much more rapidly. Water is a catalyst both for the formation of H atoms from  $H_2$  molecules and for the formation of  $H_2$  molecules from H atoms. A hydrogen-replacing metal, e.g. iron, decomposes  $H_2O$  adsorbed on its surface:



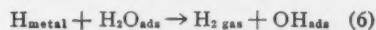
This reaction may be reversed:



The two reactions (3) and (4) will be assumed to have zero activation energy, implying that there is no change in bond energy in those reactions. The  $OH_{ads}$  formed in (3) decomposes  $H_2$ :



Similarly,  $H_2O_{ads}$  promotes the desorption of H from the metal:



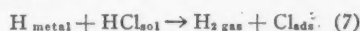
The rates of (5) and (6) determine the rates of adsorption and desorption, respectively, and the boundary conditions for the absorption-desorption process.

Decomposition and recombination may be accelerated by other catalysts than  $H_2O$ , e.g.  $H_2S$ . But, since  $H_2O$  is always present to some extent on metals and in the atmosphere under ordinary experimental conditions,  $H_2O$  is a particularly important catalyst.

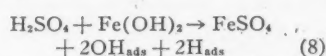
In principle, reaction (5) may also occur as a homogeneous reaction with OH radicals in the gas phase. But such OH radicals will also engage in other reactions, e.g. on the walls of the vessel containing the gas. The homogeneous decomposition of  $H_2$  assumably contributes insignificantly to the supply of H atoms as compared to the heterogeneous reaction (5). The presence of OH radicals in the gas phase will be disregarded.

In principle there is little difference between absorption-desorption mechanisms in dilute acid solution and moist  $H_2$  gas. A piece of iron immersed in a dilute solution of  $H_2SO_4$ , for example, reacts primarily with the solvent  $H_2O$ , which adsorbs and decomposes according to reaction (3). The role of the acid is to dissolve the hydroxide formed in that reaction. This is a slow process with a sufficiently dilute acid.

There are cases of a considerably different mechanism. Thus, HCl removes H from Fe and Al containing Si by the reaction<sup>8</sup>



and  $HNO_3$  removes H from Fe, for example, by the same mechanism even in the absence of a catalyst.<sup>9</sup> This does not apply to  $H_2SO_4$ , however. In dilute  $H_2SO_4$  solutions, H is desorbed by reaction (6). Absorption of H occurs by reaction (3) and in the reaction of the acid with the hydroxide:



Since the amount of metal dissolved is assumed to be negligible, the reaction (8) consists effectively of the supply of two H atoms to the surface for each  $H_2SO_4$  molecule reacting. The net result is the catalytic decomposition of  $H_2SO_4$  by  $OH_{ads}$  to give  $2H_{ads}$ . The reactions (5) and (8) are therefore similar, effectively identical.

In order to take part in reaction (5), the  $H_2$  molecule must arrive at the metal surface. According to the kinetic theory of gases, the number of  $H_2$  molecules impinging on unit surface area in unit time is

$$k_1(H_2) = \sqrt{\frac{RT}{2\pi M}} (H_2) \quad (9)$$

when  $M$  is the mass of the  $H_2$  molecule and  $(H_2)$  is the number of  $H_2$  molecules in unit volume of the gas. If an  $H_2$  molecule hits an empty adsorption site, it will become adsorbed on the surface. In that position the  $H_2$  molecule may react according to reaction (5) or, if there is no  $OH_{ads}$  available, it will desorb. If the  $H_2$  molecule hits a site adjacent to one occupied by  $OH_{ads}$ , reaction (5) occurs instantly; if

there is no adjacent  $OH_{ads}$ , the  $H_2$  molecule is instantly desorbed.

Since the metal surface is a source (in desorption) or a sink (in absorption) of  $H_2$  molecules, the distribution in space of  $H_2$  molecules cannot be perfectly uniform. The rates of absorption and desorption are small compared to the rate of redistribution in the gas. The deviation of the actual distribution from the uniform distribution is then negligible, and  $(H_2)$  is the average concentration of  $H_2$  molecules in the gas phase.

This applies also to  $H_2O$  in the vapor phase. The rate of arrival of  $H_2O$  molecules at the metal surface is

$$\sqrt{\frac{RT}{2\pi M}} (H_2O) \quad (10)$$

when  $M$  denotes the mass of the  $H_2O$  molecule.

In the case of liquid  $H_2O$ , as in absorption of H from an acid solution, the same rate formula (10) holds. But in order to become adsorbed on the surface, the  $H_2O$  molecule must break the bonds by which it is held in the liquid. Therefore, of all the  $H_2O$  molecules arriving at the surface, only

the fraction  $e^{-\frac{\Delta E}{RT}}$  can be adsorbed,  $\Delta E$  denoting the bond energy, i.e. the heat of vaporization of  $H_2O$ . In a dilute solution  $\Delta E = 10.6$  kcal/mol just as for pure  $H_2O$ . The rate of adsorption of  $H_2O$  from liquid  $H_2O$  is thus

$$\sqrt{\frac{RT}{2\pi M}} (H_2O) e^{-\frac{\Delta E}{RT}} \quad (11)$$

There is no activation energy associated with migration as such, only with removal of  $H_2O$  from the solution. The validity of the formulae (10) and (11) was confirmed by measurements of the rates of dissolution of iron in  $HNO_3$ <sup>6,7</sup> and HCl.<sup>5</sup>

Similarly, in order to take part in reaction (6), the dissolved H atom must arrive at the surface. As in the case of  $H_2O$  held in solution, the rate is

$$k_2(H) = \sqrt{\frac{RT}{2\pi M}} (H) \quad (12)$$

when  $M$  is the mass of the H atom and  $(H)$  is the concentration of H in the metal. For a non-uniform distribution,  $\{H\}$  is the concentration close to the surface. If the distribution deviates little from the uniform distribution,  $\{H\}$  differs little from the average concentration. The thinner the metal, the shorter the diffusion paths, the quicker the uniformization of the distribution, and the smaller the difference between  $\{H\}$  and the average distribution. Therefore, for a sufficiently thin specimen,  $\{H\}$  may be identified with the average concentration.

In the following the metal is assumed to be thin enough, and formulae will be derived for  $\{H\}$  for such a specimen. These formulae will then be checked by comparison with observations. Agreement with observation may be taken as indication of validity of the formulae and therefore of this assumption.

Of course, in absorption without desorption, the distribution of H in the

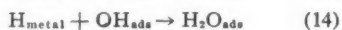
metal is irrelevant. The actual distribution is important only in connection with desorption because distribution affects the value of  $\{H\}$  to be used in formula (12).

The formula (12), like (10), implies zero activation energy for migration of H atoms in the metal. It will further be assumed that there is no activation energy for the removal of H by reaction (6). These assumptions will be justified by comparison with experimental data.

There is a conspicuous difference between reactions (5) and (6): reaction (5) leads to an increase in coverage, reaction (6) leads to no change in coverage. If  $H_{ads}$  in reaction (5) dissolves immediately, there is no change in coverage. If  $H_{ads}$  is assumed as a necessary intermediate product both in absorption and in desorption and the rate of dissolution of  $H_{ads}$  is increased to infinity, then the absorption by reactions (5) and (3) will be accelerated, but the desorption by reaction (6) will be inhibited. On the other hand, if instantaneous dissolution of  $H_{ads}$  is assumed, reaction (5) may be regarded as the heterogeneous catalyzed decomposition of  $H_2$ , requiring the adsorption of  $H_2$ , while reaction (6) may be regarded as the homogeneous (in the solid phase) destruction of dissolved H.

It is convenient, therefore, to assume a finite rate of dissolution of  $H_{ads}$  in adsorption such that a fraction  $1-\alpha$  of the H atoms formed in reaction (5) remains on the surface as  $H_{ads}$  while the fraction  $\alpha$  dissolves, and to assume an infinite rate of dissolution of  $H_{ads}$  in desorption such that the reaction of  $H_{metal}$  with  $H_2O_{ads}$  requires a direct encounter. The infinite rate of dissolution of  $H_{ads}$  in absorption is then materialized by putting  $\alpha=1$ .

Consequently, reactions (3) and (4) should be replaced by



in desorption. The rate of dissolution of  $H_{ads}$  is the rate of decomposition of the intermediately formed hydride.

The adsorbed  $H_2O$  is assumed to be always at equilibrium with the gaseous  $H_2O$ ,



Since there is no activation energy for adsorption, the activation energy associated with the equilibrium is that of desorption. This energy is  $\Delta E = 16.5$  kcal/mol according to unpublished calculations and  $\Delta E = 17$  kcal/mol according to observation.<sup>8</sup> When a crystalline hydroxide is developed, this value is reduced by the lattice strain energy which may amount to 2 to 3 kcal/mol.

#### Absorption and Desorption Kinetics

Absorption will be considered first at such low concentrations  $\{H\}$  in the metal that desorption is negligible. Reactions (3), (4) and (5) will be involved while reaction (6) does not occur. The  $H_2O$  pressure is assumed to be high enough that the metal surface is almost covered by  $H_2O_{ads}$ . The equilibrium between  $H_2O_{gas}$  and  $H_2O_{ads}$  is expressed by the formula

$$(H_2O) (1-\theta) = K[H_2O] \quad (16)$$

denoting concentrations in the gas by parentheses, coverages by square brackets, and the total coverage by  $\theta$ . The assumption just stated that  $(H_2O)$  is high enough entails the relations:

$$[H_2O] = \sim 1, 1-\theta \approx 1, [OH] \approx 1, [H] \approx 1 \quad (17)$$

Reactions (3) and (4) are assumably at a balance. The rate of decomposition of  $H_2O_{ads}$  by reaction (3) is proportional to  $[H_2O] (1-\theta)$ . Of the H atoms formed by reaction (3), the fraction  $(1-\alpha)$  remains as  $H_{ads}$ . The rate of formation of  $H_{ads}$  by reaction (3) is thus proportional to  $[H_2O] (1-\theta) (1-\alpha)$ . The rate of destruction of  $H_{ads}$  by reaction (4) is proportional to  $[H] [OH]$ . When those rates are equal, equilibrium is expressed by the formula

$$[H] [OH] = K' [H_2O] (1-\theta) (1-\alpha) \quad (18)$$

The rate of formation of  $H_{ads}$  by reaction (5) is

$$\frac{d[H]}{dt} = (1-\alpha) k_1 (H_2) [OH] (1-\theta) \quad (19)$$

Since reactions (3) and (4) cancel, equation (19) gives the total rate of formation of  $H_{ads}$ .

Solving equation (18) for  $[OH]$  and equation (16) for  $(1-\theta)$  and inserting them in equation (19) give

$$\frac{d[H]}{dt} = (1-\alpha)^2 k_1 K' K'' (H_2) \frac{[H_2O]^2}{[H] (H_2O)^2} \quad (20)$$

Integration gives with  $[H_2O] = \sim 1$

$$[H] = (1-\alpha) K \sqrt{2k_1 K'} \frac{\sqrt{(H_2)}}{(H_2O)} \sqrt{t} \quad (21)$$

For simplicity,  $[H] = 0$  is assumed for  $t = 0$ .

For a metal sheet of thickness  $d$  and unit surface area, each H atom dissolved in the metal contributes  $\frac{1}{d}$  to the average concentration  $\{H\}$  in the metal. Since only a fraction  $(1-\alpha)$  of the empty sites adjacent to  $OH_{ads}$  becomes occupied as a result of reaction (5), the  $H_2O_{ads}$  formed in reaction (5) may use the empty sites left for decomposition according to reaction (3). Therefore

$$\frac{d\{H\}}{dt} = \frac{\alpha}{d} k_1 (H_2) [OH] (1-\theta) (1+\alpha) \quad (22)$$

or

$$\frac{d\{H\}}{dt} = \frac{1}{d} \frac{\alpha(1+\alpha)}{1-\alpha} \frac{d[H]}{dt} \quad (23)$$

Inserting equation (21) in equation (23) and integrating give

$$\{H\} = \frac{\alpha(1+\alpha)}{d} K \sqrt{2k_1 K'} \frac{\sqrt{(H_2)}}{(H_2O)} \sqrt{t} \quad (24)$$

assuming  $\{H\} = 0$  for  $t = 0$ .

Since  $(1-\alpha)$  is small,  $\alpha \approx \sim 1$  may be taken. Then  $[H] = \sim 0$ ,

$$\frac{d[H]}{dt} = \sim 0 \text{ and}$$

$$\{H\} = \frac{2}{d} K \sqrt{2k_1 K'} \frac{\sqrt{(H_2)}}{(H_2O)} \sqrt{t} \quad (25)$$

Desorption in the absence of absorption is considered now. This case is materialized when  $\{H\}$  is large as compared to  $(H_2)$ . The metal has been assumed to be charged with H by absorption as discussed above and then placed in an atmosphere containing very little  $H_2$ . Reactions (3) and (4) are now replaced by reactions (13) and (14), assumed to be at a balance so that

$$\{H\} [OH] = K'' [H_2O] \quad (26)$$

The rate of desorption is

$$\frac{d\{H\}}{dt} = -\frac{k_2}{d} \{H\} [H_2O] \quad (27)$$

The general solution may be obtained with the relation

$$[H_2] + [OH] = \theta \quad (28)$$

Equations (16), (26) and (28) give

$$[H_2O] = \frac{(H_2O) \{H\}}{(H_2O) (K'' + \{H\}) + K \{H\}} \quad (29)$$

When  $\{H\}$  is small enough,  $\{H\}$  may be ignored at the side of  $K''$ . In this case

$$[H_2O] = \frac{(H_2O) \{H\}}{K'' (H_2O) + K \{H\}} \quad (30)$$

For a carefully dried gas,  $(H_2O)$  is small and

$$[H_2O] = \frac{(H_2O)}{K} \quad (31)$$

This inserted in Equation (27) yields

$$\log \{H\} = \log \{H\}_0 - \frac{k_2 (H_2O)}{d K} t \quad (32)$$

For a moist gas or liquid water,  $(H_2O)$  is large and

$$[H_2O] = \frac{\{H\}}{K''} \quad (33)$$

This inserted in (27) yields

$$\frac{1}{\{H\}} = \frac{1}{\{H\}_0} + \frac{k_2}{d K''} t \quad (34)$$

It is illuminating that  $k_2$  and  $d$  occur only in the combination  $\frac{k_2}{d}$  in the desorption formulae. This is because the rate of desorption depends on the rate of arrival of H atoms at the surface. This rate is determined by the ratio of migration velocity and migration path length, i.e. by  $\frac{k_2}{d}$ .

The rate of desorption in terms of  $H_2$  gas evolved is

$$\frac{d(H_2)}{dt} = k_2 \{H\} [H_2O] = -d \frac{d\{H\}}{dt} \quad (35)$$

The rate depends thus by a factor  $d$  on whether it pertains to  $H_2$  gas evolution or H concentration, i.e. to amount of H desorbed or to reduction of concentration.

The case when absorption and desorption occur simultaneously and at equal rates is of interest because it determines the saturation level of the H concentration in the metal:



$$\frac{d\{H\}}{dt} = \frac{2}{d} k_1 (H_2) [OH] (1 - \theta) - \frac{1}{d} k_2 \{H\} [H_2O] = 0 \quad (36)$$

Eliminating  $[OH]$  by means of equation (26) and  $(1 - \theta)$  by means of equation (16) gives

$$\{H\}^2 = 2 \frac{k_1}{k_2} K K'' \frac{[H_2O]}{(H_2O)} (H_2) \quad (37)$$

When  $\{H\}$  is large, Equation (29) takes the form

$$[H_2O] = \frac{(H_2O)}{K + (H_2O)} \quad (38)$$

This inserted in Equation (37) yields

$$\{H\}^2 = 2 \frac{k_1}{k_2} K K'' \frac{(H_2)}{K + (H_2O)} \quad (39)$$

In a dry atmosphere,  $(H_2O)$  small, this gives

$$\{H\} = \sqrt{2 \frac{k_1}{k_2} K''} \sqrt{(H_2)} \quad (40)$$

In a wet atmosphere,  $(H_2O)$  large, it gives

$$\{H\} = \sqrt{2 \frac{k_1}{k_2} K'' K} \frac{\sqrt{(H_2)}}{\sqrt{(H_2O)}} \quad (41)$$

In both cases  $\{H\}$  is independent of  $d$ . The formulae (40) and (41) are therefore not limited to thin specimens. This is obvious since, at saturation, the net rate of the surface processes is zero. Therefore, however small the diffusion constant, the concentration gradient will also be zero at saturation. The assumption of a uniform distribution must therefore hold strictly at saturation.

The formulae may be simplified by inserting the values of  $k_1$  and  $k_2$  according to formulae (9) and (12), for

$$\frac{k_1}{k_2} = \frac{1}{\sqrt{2}} \quad (42)$$

Simultaneous absorption and desorption are also encountered in the permeation of membranes by hydrogen. A sheet of metal is exposed to  $H_2$  gas on one side and to a different gas on the other side. The case of wet  $H_2$  gas on one side and a wet atmosphere free of  $H_2$  on the other side will be considered. Conditions will be assumed such that desorption at the absorption side and absorption at the desorption side may be ignored.

After some time, a stationary state will be established when the rates of absorption and desorption are equal. Then, with  $\{H\}$  according to (25) and  $[H_2O] = 1$  on the desorption side,

$$\frac{d\{H\}}{dt} = \frac{1}{d} K \sqrt{2k_1 K'} \frac{\sqrt{(H_2)}}{(H_2O)} \frac{1}{\sqrt{t}} - k_2 \frac{2}{d} K \sqrt{2k_1 K'} \frac{\sqrt{(H_2)}}{(H_2O)} \sqrt{t} = 0 \quad (43)$$

This gives

$$t = \frac{d}{2k_2} \quad (44)$$

and the rate of desorption,

$$k_2 \{H\} = \frac{2}{d} K \sqrt{k_1 k_2 K'} \frac{\sqrt{(H_2)}}{(H_2O)} \quad (45)$$

in the stationary state.

In the case of a dilute  $H_2SO_4$  solution

instead of moist  $H_2$  gas, the results arrived at for moist  $H_2$  gas may be taken over, merely replacing  $(H_2)$  by  $(H_2SO_4)$  and giving new values to the constants.

The values of  $K'$  and  $k_2$  are not changed. The value of  $k_1$  is changed to

$$k_1 = \sqrt{\frac{RT}{2\pi M}} \frac{1}{H_2SO_4} \quad (46)$$

The value of  $K$  is different since there is an equilibrium of adsorbed  $H_2O$  with liquid  $H_2O$ . The desorption of  $H_2O_{ads}$  has the same activation energy, but the adsorption now has an activation energy of 10.6 kcal/mol, the heat of vaporization of liquid water. The difference, 5.9 kcal/mol, is the apparent activation energy associated with the new equilibrium constant. If the possibility of desorption from crystalline hydroxide is again admitted, this value may be reduced to about 3 or 4 kcal/mol.

#### Comparison With Observations

The purpose of this section is to check the premises, on which the deductions in the preceding section were based, as to their validity under ordinary experimental conditions. In order to do so, it may be appropriate to start out by summarizing those premises.

In the treatment of absorption, no desorption is assumed to occur simultaneously. The distribution of  $H$  atoms in the metal, whether uniform or not, is irrelevant as long as the concentration close to the surface is low enough to make desorption negligible. This assumption is valid for small values of  $t$ , and the larger the diffusion constant  $D$ , the larger the range of  $t$ .

In the treatment of solubility a uniform distribution of  $H$  in the metal has been assumed. Since, at equilibrium, the net rate of the surface processes (absorption and desorption) is zero, this assumption is valid for any value of the diffusion constant  $D > 0$ .

In the treatment of desorption and permeation  $D$  is assumed to be large enough and the surface processes slow enough to maintain almost uniform distribution of  $H$  in the metal. This assumption is valid for sufficiently thin specimens. The larger  $D$  and the slower the surface processes, the larger is the range of  $d$  for which it is valid.

Of particular interest is the range of  $d$ , for which this assumption is valid under ordinary conditions.

The rate constant  $k_2$  is assumed to be associated with zero activation energy. This assumption does not affect the validity of the formulae containing  $k_2$ . It will be checked by comparison with observed activation energies.

The formula

$$\{H\} = \text{const} \times \sqrt{(H_2)} \quad (47)$$

is known as Sievert's law and was confirmed by Naumann<sup>9</sup> up to 1000 atmospheres of  $H_2$  pressure. According to formula (40), the apparent activation energy associated with the solubility is  $\frac{1}{2} \Delta E$ , when  $\Delta E$  is the heat of desorption of  $H_2O$ . Since  $\Delta E = 16.5$  kcal/mol for adsorbed  $H_2O$  and may be reduced to  $\Delta E = 13.5$  kcal/mol for a crystalline hydroxide, this statement can be given:

$$6.75 < \frac{1}{2} \Delta E \leq 8.25 \text{ kcal/mol} \quad (48)$$

Observed values are 6.7 kcal/mol for the solubility in solid  $Fe^0$  and 8.0 kcal in liquid  $Fe^0$ . The activation energy for the dissolution of  $Al$  in  $HCl$  solutions in the range 6-10 N, where the removal of  $H$  is rate-determining, was found to be 6.87 kcal/mol.<sup>5</sup> Hence, within the experimental accuracy,  $k_2$  and therefore migration of  $H$  atoms in the metal are associated with zero activation energy.

The formula (25) for adsorption may be written

$$\{H\} = \frac{\text{const.}}{d} \sqrt{(H_2)} \sqrt{t} e^{-\frac{\Delta E}{RT}} \quad (49)$$

with

$$13.5 < \Delta E \leq 16.5 \text{ kcal/mol} \quad (50)$$

That  $\{H\}$  is proportional to  $\sqrt{t}$  has been found by a number of investigators, e.g., Darken and Smith,<sup>23</sup> for absorption from dilute  $H_2SO_4$  solution.

Activation energies for absorption from  $H_2$  in the range or formula (50) have been reported by a number of investigators, e.g. 14.6 kcal/mol for absorption by Pyrex glass,<sup>24</sup> 14.0<sup>25</sup> and 14.1<sup>26</sup> kcal/mol for absorption by  $Cu$ ,  $16 \pm 1$  kcal/mol for adsorption on  $Ni$  catalyst,<sup>27</sup> and 16.1 kcal/mol for adsorption on  $V_2O_5$ .<sup>28</sup> Since the activation energy is that for desorption of  $H_2O_{ads}$ , it is encountered in many instances where this process is rate-determining, not only in connection with absorption of hydrogen by metals. On the other hand, the activation energy for absorption of  $H$  may differ from formula (50) because another mechanism is operative. Thus, the observed activation energy may be that for the reduction of an oxide on the metal. This may be expected to apply to permeation measurements when a membrane is exposed to  $H_2$  on one side and to an oxidizing atmosphere on the other side. Permeation measurements therefore give an ambiguous check on the activation energy. Values observed are 16.5, 17.0, and 15.3 kcal/mol for the permeation of  $Fe-Mo$  alloys of 0.98, 8.36, and 10.36 percent  $Mo$ , respectively,<sup>29</sup> and 17.4 kcal/mol for the permeation of  $Cu$ .<sup>30</sup>

Observations as to the effect of  $d$  on the rate of absorption are scarce. The data of Darken and Smith<sup>23</sup> indicate proportionality to  $\frac{1}{d}$  in agreement with formula (25). Recently pertinent data were presented by Butler and Davis.<sup>31</sup> The experimental set up was similar to that of Aten and Zieren,<sup>21</sup> Fischer and Heiling,<sup>22</sup> and Bagotskaya and Frumkin.<sup>23</sup> A steel membrane is exposed to a  $NaCr_2O_7$  solution on one side and to a  $H_2SO_4$  solution on the other side. The potential on the dichromate side is recorded. When the potential has reached a constant value in the absence of  $H_2SO_4$ , the acid is added and the potential change with time recorded. The time  $t_0$  elapsed between addition of acid and change of potential was found to be proportional to  $d^3$  up to 1-inch thickness of the membrane. In this experiment the dichromate side of the membrane was originally covered with  $O$  or oxide. Thus it had the  $O$  potential. The permeating  $H$  combines with the  $O$  and changes the potential to that of  $OH$  and eventually to that of  $H_2$ . The change in potential occurs when the concentration  $\{H\}$  at the dichromate side has reached a certain level  $\{H\}_0$ . Inserting  $\{H\}_0$  for  $\{H\}$  in (49) shows that  $t_0$  is proportional to  $d^3$  in agreement with observation.

The rate of permeation is frequently held to be proportional to  $\frac{1}{d}$ . It appears that few investigators give data in support of this view. It seems to be based mainly on the results of Lewkonja and Baukloh.<sup>24</sup> These investigators charged with H<sub>2</sub> gas in the range 700-1000 C and measured the volume of H<sub>2</sub> gas on the desorption side. The data were not analyzed as published and indicate that the rate is proportional to  $\frac{1}{\sqrt{d}}$  at 800

C and to  $\frac{1}{d}$  at 980 C. Most of the H absorbed at the higher temperature was probably produced by a homogeneous gas phase reaction. This would make the rate proportional to  $\frac{1}{d}$  and also to (H<sub>2</sub>) instead of to  $\frac{1}{\sqrt{d}}$  and  $\sqrt{(H_2)}$ . Unfortunately, the dependence on the H<sub>2</sub> pressure was not determined. At lower temperatures, the heterogeneous formation of H should predominate and the rate be proportional to  $\frac{1}{\sqrt{d}}$ .

The same argument is applicable to electrolytic charging with hydrogen, which is equivalent to the homogeneous formation of H atoms. Aten and Zieren<sup>25</sup> found with electrolytic charging that the rate of permeation was not a linear function of  $\frac{1}{d}$ . Heath,<sup>25</sup> also charging electrolytically, found a linear relation with  $\frac{1}{d}$  for  $d < 0.7$  mm only; for thick membranes the curve given by Heath looks more like a linear function of  $\frac{1}{d^2}$ . In electrolytic charging, additional phenomena may occur since not only H is formed at the surface.

Desorption rate measurements are scarce. Andrews, et al.,<sup>3</sup> made an extensive study of desorption rates at 400 to 1100 C from steel immersed in molten salt or molten lead. The formula (32) would be expected to apply to those conditions. Actually a combination of an exponential decay and one of the form (34) was found to fit the data best. Clearly, a formula of the form (29) may be approximated by a variety of functions depending on the ranges of the variables and on the values of the rate and equilibrium constants.

Formula (32) gives desorption rates which decrease with increasing temperature. The initial rate according to (32)

$$\text{is } \frac{d(H_2)}{dt} = -d \frac{d(H)}{dt} = \{H\}_s \frac{k_2(H_2O)}{K} \quad (51)$$

where

$$K = K_0 e^{-\frac{\Delta E}{RT}} \quad (52)$$

and  $\Delta E = 16.5$  kcal/mol or slightly less.

Eisenkolb and Ehrlich<sup>26</sup> found that the time for complete H<sub>2</sub> evolution from a charged austenitic steel was increased from 1 hour at room temperature to 3 hours by raising the temperature to 140 C. This indicates a smaller value of  $\Delta E$ , however.

Desorption at a constant rate has been reported by Bénard and Albert<sup>27</sup> for Pd foils in an oxidizing solution and by Andrew, et al.,<sup>3</sup> for comparatively thick specimens ( $\frac{1}{4}$ -inch diameter) of a 3.4 percent chromium steel immersed in molten lead at 700 C. Both Pd and Cr form hydrides. The desorption formulae are therefore not applicable to those cases. However, an important conclusion may be drawn from those observations.

A constant rate of desorption, independent of the concentration of H in the metal, indicates a constant concentration of H at the surface and a rapid enough diffusion of H in the metal to maintain that concentration. In the experiment of Bénard and Albert,<sup>27</sup> the Pd foil was charged far above the hydride composition. In the experiment of Andrew, et al.,<sup>3</sup> it is conceivable that chromium hydride was formed at spots on the surface and that desorption occurred via that hydride. In both cases the diffusion of H in the metal was rapid enough to reform the compound at the rate it was decomposed by desorption.

These results, whatever interpretation is given to the details of the mechanism, show conclusively that the rate of diffusion was high enough not to affect the rate of desorption under these experimental conditions.

### Conclusions

Desorption is accelerated by water. Thus, steel gives off its hydrogen much more rapidly when immersed in water than when held in air. Hydrogen is more rapidly absorbed from moist than from dry H<sub>2</sub> gas. Similarly, absorption from acid solutions is accelerated by As<sub>2</sub>O<sub>3</sub>, HgCl<sub>2</sub> and other additions to the solution.

Additions to the source of hydrogen cannot be expected to affect the rate of diffusion of H in the interior of the metal. They can affect only the surface processes by which H atoms are supplied to or removed from the surface of the metal. Therefore, under ordinary experimental conditions, particularly in the absence of such catalysts, these surface processes are slow.

It follows from these qualitative observations and from the desorption experiments referred to that, under ordinary experimental conditions, and particularly under the conditions considered in this paper, diffusion in the metal is rapid as compared to the surface processes. The distribution of H in the metal during absorption and desorption can be assumed to be almost uniform up to at least 1-inch thickness.

Furthermore, comparing the formulae

derived for absorption, solubility, and permeation with observations, agreement is reached as to the dependence on H<sub>2</sub> gas pressure, time, thickness of the specimen, and temperature. In particular, the observed activation energies agree within the experimental accuracy, with those calculated. Within the experimental accuracy, the migration of H in the metal is associated with zero activation energy.

NOTE: Since this paper was prepared early in 1956, Hudson et al.<sup>28,29</sup> have reported particularly pertinent experimental investigations of the role of water in the desorption of hydrogen from steel. In particular, empirical formulae of the forms (32) and (34) were derived from the data for dried and moist atmospheres, respectively.

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Any discussions of this article not published above will appear in the June, 1959 issue

# Evaluation of Refinery Corrosion Inhibitors\*

By A. J. FREEDMAN and A. DRAVNIKS

## Introduction

USE OF inhibitors to suppress internal corrosion in pipelines, process units and tankage has become widespread. The total cost of corrosion to the oil-refining industry is approaching \$300,000,000 annually. This cost would be considerably higher if inhibitors were not used to control corrosion rates in critical refinery equipment. Unfortunately, inhibitors vary widely in their efficiency and in their response to different process conditions. Because of the savings which can be achieved by decreasing the cost of corrosion, the proper inhibitor must be chosen for each application. Selection of the best compounds from the large number of inhibitors commercially available is a difficult task. Also, money spent to develop new corrosion inhibitors probably is not worthwhile unless it can be shown that the compounds in use at present are either too expensive or not effective in a given application.

This paper presents a discussion of differences in the behavior of refinery corrosion inhibitors under various process conditions. New laboratory testing procedures are described that can be used to evaluate the response of inhibitors to specific operating variables.

## Laboratory vs Refinery Evaluations

Table 1 shows some comparative corrosion rates obtained in the laboratory with five different organic corrosion inhibitors. Both water-soluble and oil-soluble types are included. The data were obtained by the electrical resistance method using equipment described previously for evaluating rust inhibitors for petroleum products.<sup>1</sup> The corrosive medium in these tests was a stirred mixture of virgin light naphtha plus 1 percent of an aqueous phase containing 1 percent ammonium chloride. The water phase was adjusted to various pH levels with ammonia and hydrochloric acid. The inhibitors were added at a concentration of 20 ppm based on the total liquid volume. At a pH of 8, inhibitors E and F are not effective, inhibitor C provides moderate protection, and inhibitors A and G are very good. At a pH of 3, the corrosion rate with no inhibitor is much higher. Under these severe conditions, compound E again is of no value, and compound C is only partially effective. Inhibitor A, however, now seems to be less useful than inhibitor C. In contrast to this behavior, inhibitor F is effective under acid conditions, whereas at a pH of 8 it shows little, if any, corrosion protection. The effectiveness of compound G is not disturbed by pH changes. This inhibitor produces excellent results at both low and high pH.

These data show that corrosion



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inhibitors respond differently to pH changes in the test environment. In Table 2 are shown corrosion rates, measured while using three of these same inhibitors, in the overhead system of a crude distillation unit. The data were obtained with electrical resistance corrosion probes,<sup>2</sup> at a pH of 8 and an inhibitor concentration of 20 ppm. These results agree with the laboratory data at a pH of 8. Money spent on inhibitor E in this location would be wasted. Inhibitor C, although it offers some protection, would be much less effective than compound A. In this case, inhibitor A is intermediate in cost between the other two. Further tests showed that because inhibitor A is so effective, it can be used at lower concentrations than inhibitor C. Hence, of the compounds tested, inhibitor A is both the most effective and the most economical material for use in this location.

Refinery corrosion inhibitors should be chosen carefully to match each application. Ideally, this should be done by evaluating a series of compounds under actual plant conditions. However, many independent, noncontrollable variables influence the course of normal plant operations. These variables make quantitative testing under refinery conditions a difficult, time-consuming and expensive operation. Some type of laboratory screening procedure is required to select the most effective inhibitors for field trials.

## Abstract

Since reproduction of refinery operating conditions in the laboratory is difficult, most laboratory testing of refinery corrosion inhibitors has been unreliable. For this reason, inhibitors for refinery use have been selected mainly on the basis of qualitative field trials. Such tests are expensive and subject to many independent, noncontrollable variables.

Frequently, failures of corrosion inhibitors to provide satisfactory protection can be traced to specific environmental factors. Therefore, new laboratory tests for commercial refinery corrosion inhibitors have been developed to simulate closely certain features of actual field conditions. Inhibitors have been evaluated for corrosion protection in light naphtha condensers, distillation towers, special high temperature locations, high velocity service, narrow crevices, and for impingement attack.

These test data show conclusively that no single corrosion inhibitor can be expected to provide satisfactory corrosion protection in all refinery locations. Plant results obtained with electrical resistance corrosion probes corroborate the laboratory evaluations. 5.8.1

Laboratory evaluations of refinery inhibitors have been open to question in the past because of the difficulties involved in reproducing actual refinery conditions under which an inhibitor must provide protection. There is, however, an alternative approach to this problem. Table 3 lists some of the important factors which can influence the performance of a corrosion inhibitor in a plant situation. The corrosion rate in a given location depends upon both the composition of the stream and the operating conditions on the unit in ques-

TABLE 1—Effects of pH on Corrosion Rate Laboratory Data\*

Inhibitor	Corrosion Rate (IPY)	
	pH = 8	pH = 3
None.....	0.060	0.510
A.....	0.002	0.260
C.....	0.008	0.110
E.....	0.060	0.480
F.....	0.040	0.004
G.....	0.000	0.001

\* Test conditions: Virgin light naphtha plus 1% aqueous  $\text{NH}_4\text{Cl}-\text{HCl}$ . Rapid stirring, air present. T = 104 F. Data obtained by the electrical resistance method. Inhibitor concentration = 20 ppm.

TABLE 2—Effects of Inhibitors on Corrosion Rates in Crude Distillation Overhead System\*

Inhibitor	Corrosion Rate (IPY**)
None.....	.110
A.....	.001
C.....	.010
E.....	.080

\* Inhibitor concentration = 20 ppm; pH = 8

\*\* Data obtained with electrical resistance corrosion probes.

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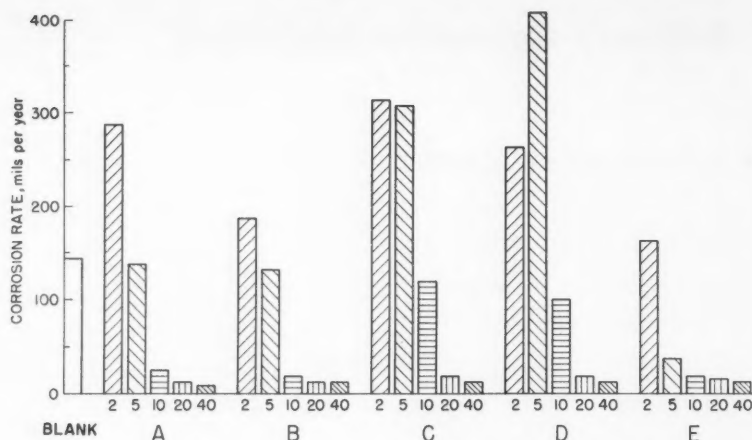


Figure 1—Effect of concentration on corrosion inhibitor performance in the presence of hydrogen sulfide.

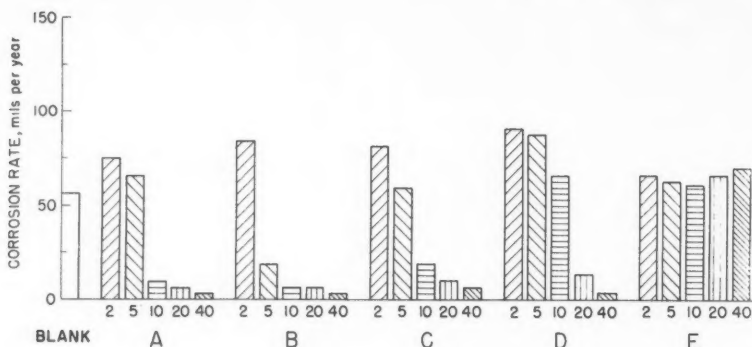


Figure 2—Effect of concentration on corrosion inhibitor performance in the presence of air.

tion. Each of these overall variables can be separated into a number of single factors as shown in Table 3. Laboratory tests have been devised to measure quantitatively the response of corrosion inhibitors to each of these variables under standardized conditions. These conditions do not necessarily represent the actual operations of any particular refinery unit. The results of these tests, however, provide a reliable scale for relative comparisons of many different inhibitors. The data can be used to select those compounds for field trials most likely to succeed in a given situation and to shorten the time required for these trials.

#### Composition Variables

##### Hydrogen Sulfide

The composition variables listed in Table 3 can be evaluated readily in an electrical resistance assembly of the type described previously.<sup>1</sup> Figure 1 summarizes the results of a series of dynamic tests conducted with this apparatus to measure the performance of inhibitors in a hydrogen sulfide atmosphere. In this graph, the corrosion rate is shown on the vertical axis, and the shaded bars indicate rates obtained with increasing concentrations of various inhibitors, in parts per million. The white bar on the left represents the corrosion rate with no inhibitor. The corrosive medium in these tests was a stirred mixture of virgin light naphtha and tap water adjusted to a pH of 6.5. The atmosphere consisted of one-half volume percent of

hydrogen sulfide in hydrogen. All data were collected at 104 F.

The five inhibitors shown in Figure 1 behave differently in this environment. In every case, the corrosion rates at low inhibitor concentrations are higher than those with no inhibitor. The amount of increase in corrosion rate and the inhibitor concentration required to establish normal protection vary with each material. The more powerful inhibitors in this environment, such as compounds A and B, become effective at concentrations between 5 and 10 ppm. With other inhibitors, the initial rise in corrosion rate is more pronounced, and additions of greater than 10 ppm are required to establish positive protection. This corrosion rate increase at low inhibitor concentrations actually has been observed in the field with the aid of electrical resistance corrosion probes. Care should be taken in all applications that enough inhibitor is used to avoid this problem. About 5 ppm should be sufficient in most cases.

##### Oxygen

In the presence of hydrogen sulfide, all the inhibitors tested show good results, if their concentration is sufficiently high. In the presence of air, however, this is not the case. Figure 2 presents a series of test data obtained with air substituted for the hydrogen sulfide in hydrogen atmosphere. All corrosion rates are lower in this case, and the initial rise in corrosion rate is less apparent. However, in the presence of air, some inhibitors, such as E, pro-

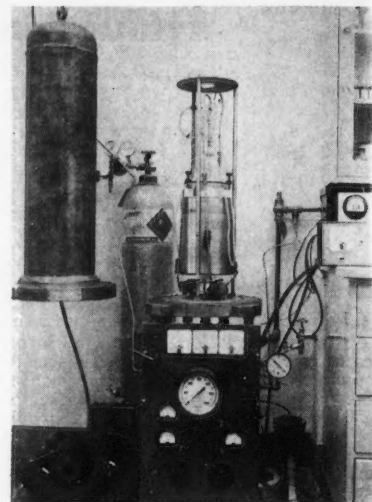


Figure 3—Pressurized laboratory test reactor.

TABLE 3—Major Environmental Factors Influencing Refinery Corrosion Rates

Composition Variables	Operation Variables
General composition.....	Temperature
Presence of oxygen.....	Fluid velocity
Presence of hydrogen sulfide.....	Impingement
pH of aqueous phase.....	Presence of crevices
	Metal composition

vide no corrosion protection at concentrations up to 40 ppm, whereas compounds A and B, for example, are effective at 10 ppm. These differences in behavior, caused by the presence of oxygen, are important. Some manufacturers claim that small concentrations of oxygen are sufficient to reduce the effectiveness of their compounds. Laboratory tests have shown that the results in Figure 2 remain valid even when the oxygen concentration in the atmosphere is reduced to as little as 10 ppm by volume. Analyses indicate that the overhead stream on a crude distillation tower may contain from 5 to 7 ppm of dissolved oxygen. In naphtha held in tankage for several days without a nitrogen blanket, the oxygen concentration may rise to over 60 ppm.<sup>2</sup> Thus, it appears that inhibitors which show extreme sensitivity to oxygen, as seen in Figure 2, should not be applied whenever any possibility exists that even low concentrations of dissolved oxygen may be present.

#### Operation Variables

##### Temperature and Velocity

Temperature limitations probably cause more difficulty in laboratory testing of inhibitors than any other single factor. For aqueous condensate type corrosion below about 200 F, the same apparatus described above for the study of composition variables is satisfactory. At higher temperatures, however, the problem is more complicated. Usually, high temperature corrosion is associated with some other operation variable, such as high flow velocity, impingement, or reflux conditions. Hence, any high temperature laboratory test apparatus should permit simultaneous and independent control of as many variables as possible so that inhibitor response

to each variable singly and in combination with others can be determined.

Figure 3 is a photograph of a pressurized laboratory reactor designed for a variety of test conditions. For maximum flexibility and corrosion resistance, the reaction equipment is constructed of glass. The heating elements and glass reactor are placed inside a metal vacuum jacket to conserve heat. A reflux condenser, an electrical resistance corrosion probe, a coupon rack and thermocouples to indicate temperatures at various points in the system are inserted through glass joints on the top flange. For velocity tests, an explosion proof motor is mounted above the condenser with a shaft extending into the reactor through a specially designed packing gland. Corrosion specimens can be mounted directly on this shaft. Cooling water connections and electrical leads are passed through the bottom plate, and the entire assembly is covered by a steel bell. The reactor then can be brought to any pressure needed to reach the desired temperature, up to limits of 500 psig and 500 F. Reducing, oxidizing, or inert atmospheres can be used. The instruments on the control panel indicate pressure, vacuum in the insulating jacket, heater and stirrer wattages, and temperatures in the reactor. A safety control is adjusted to turn off the power if temperatures in the condenser exceed a set limit.

Carbon steel velocity coupons that were exposed in the pressurized reactor are shown in Figure 4. The corrosive medium in these tests was virgin light naphtha plus 1 percent deionized water in a nitrogen atmosphere. The temperature was 350 F, and the velocity at the outer edge of the specimen in each case was 18.8 feet per second. The specimens were exposed to a mixture of liquid plus entrained vapor. With no inhibitor present, a corrosion rate of 0.025 inches per year was observed by weight loss. In addition, general and uniform pitting occurred over the entire surface of the coupon. The leading edges of the coupon at the point of highest velocity show greater attack than was found on other portions of the surface.

Addition of inhibitor A at 10 parts per million caused a definite change in the type of attack which occurred. The general corrosion rate was reduced by about 60 percent, and pitting was confined to the areas of lowest velocity, i.e., those nearest to the center. No effects of velocity on the leading edges of the specimen are apparent. The entire surface is covered by a hard, adherent coating of magnetite ( $\text{Fe}_3\text{O}_4$ ), plus organic compounds and sulfur in the form of sulfides. Inhibitor C showed different behavior. At the same concentration as inhibitor A, compound C provided tenfold better protection. General pitting was reduced on this specimen. However, the obvious velocity effect may be accentuated over that found in the blank. Each of these three tests was run twice, and the effects shown here were found to be reproducible. Thus, inhibitors A and C respond differently to the combined effects of temperature and velocity. The best choice of inhibitor for field trials will depend upon the relative importance of these two variables in the unit to be protected.

#### Impingement Attack

A particularly severe form of corrosion has been observed in refinery heat exchangers in which partially vaporized streams of corrosive fluids strike hot

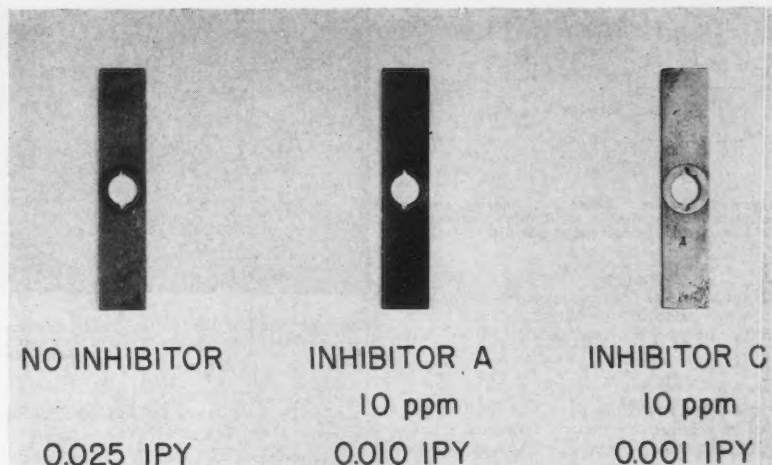


Figure 4—Effect of inhibitors on high velocity corrosion at 350 F. Test solution: virgin light naphtha plus 1 percent deionized water. The atmosphere was nitrogen; the temperature 350 F. Velocity at the outer edge of the specimen was 18.8 feet per second. Corrosion rates were measured by weight loss.

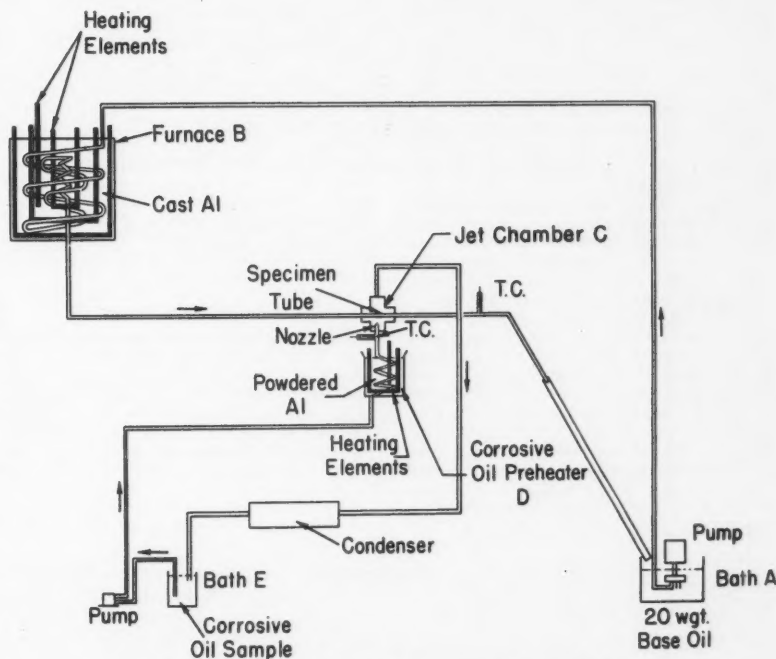


Figure 5—Laboratory equipment for impingement corrosion tests.

exchanger tubes at high velocities as in reboilers. This impingement attack is sensitive to temperature, velocity, geometry of the system and, of course, to stream composition. In Figure 5 is shown laboratory equipment designed for study of this effect under controlled conditions. To simulate the hot exchanger, 20W base oil is pumped from bath A through furnace B to the specimen tube in jet chamber C. From C the oil flows back to bath A. Furnace B consists of stainless steel tubing wound around electrical heating elements and imbedded in cast aluminum. The corrosive oil under test is pumped from bath E through preheater D to a jet nozzle. The stream then strikes the specimen tube at a 90-degree angle. Leaving the jet chamber, the corrosive stream is con-

densed and returned to supply bath E. With this equipment, the temperature of the specimen tube, the corrosive jet and the jet velocity can be varied independently to cover a wide range of test conditions.

Table 4 presents some typical results which were obtained with this equipment using an oil containing unsaturated hydrocarbons from a petrochemical process. Small amounts of corrosive organic acids and peroxides were formed in this liquid by oxidation during storage. At an oil temperature of 270 F, the corrosion rate was sensitive to the temperature of the steel specimen tube. Neutralization of the stream with potassium hydroxide reduced the corrosion rate. Inhibitor H at 40 ppm was not useful. Other inhibitors might have pro-

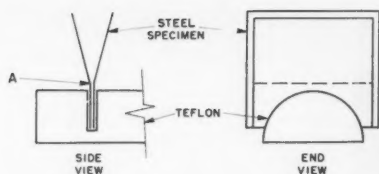


Figure 6—Specimen mount for studying crevice corrosion in inhibited acids. Crevice corrosion is most pronounced at point A.

vided good corrosion protection in this stream. In this particular case, however, it was feasible to eliminate the difficulty by preventing oxidation of the stream.

#### Crevice Corrosion

Another example of a special type of refinery corrosion which requires a specific laboratory test procedure is the problem of crevice corrosion. Crevice corrosion occurs in narrow spaces such as those found in valve bodies where normal circulation is not sufficient to prevent accumulation of corrosion products or depletion of inhibitor. A simple laboratory device for studying this problem at low temperatures is shown in Figure 6. A thin slot is cut into a half-round Teflon block, as shown, and two thin sheets of metal are forced into this slot, thus creating a narrow crevice. The entire assembly then is submerged in a beaker containing the corrosive liquid under test. This beaker can be heated, stirred or shaken to simulate any desired flow conditions.

Figure 7 shows specimens of Type 410 stainless steel tested with this apparatus in a commercial inhibited refinery cleaning acid at 140 F. In all of these tests, attack on the main surface of the metal was negligible. However, on the specimen marked Blank, the outline of the crevice in which deep corrosion occurred is clearly visible. The corrosion inhibitor incorporated in the cleaning acid was unable to prevent this attack. However, 0.3 percent additions of lead acetate or stannous chloride eliminated crevice corrosion. Addition of chloroplatinic acid, on the other hand, actually accelerated

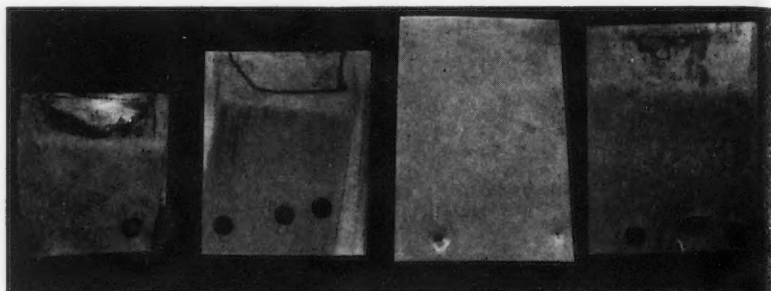


Figure 7—Effects of additives on crevice corrosion of Type 410 stainless steel in refinery cleaning acid at 140 F. The additives used on the specimens were from left to right: lead acetate, stannous chloride, blank and chloroplatinic acid.

TABLE 4—Factors Influencing Impingement Attack of Corrosive Oils on Steel

# Treatment	Oil Temp. Degrees F	Specimen Tube Temp. Degrees F	Corrosion, Rate, IPY
Raw stream.....	270	300 500 570	0.9 4.0 6.5
Stream Neutralized with KOH.....	270	500	0.3
Stream plus 40 ppm Inhibitor H.....	270	500	4.0

the attack at a concentration of only 0.06 percent. Unfortunately, when these tests were repeated in the presence of hydrogen sulfide, the tin and lead salts had no beneficial effects. Although these salts might be helpful in acid cleaning new units, they would not be useful in older equipment where sulfidic scales are present. Hydrogen sulfide probably acts by precipitating the lead and tin as insoluble sulfides.

#### Summary

Extensive laboratory work has shown that it is difficult to reproduce in the laboratory a complete set of refinery operating conditions. Single variables, either independently or in combination with other variables, can be studied under standardized test conditions. By using this approach, refinery corrosion

inhibitors were evaluated for many different types of service. No single inhibitor was found to be useful under all test conditions. Inhibitors were found to vary markedly in behavior in different environments. With the aid of standardized tests such as those described above, the most likely corrosion inhibitors can be selected quickly for further laboratory testing under more closely simulated plant conditions or for actual field trials.

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Any discussions of this article not published above will appear in the June, 1959 issue



# Corrosion of Refinery Equipment During Acid Cleaning\*

By K. R. WALSTON and A. DRAYNIEKS

## Introduction

THE USE of inhibited hydrochloric acid for the removal of fouling deposits from refinery equipment has gained considerable prominence during the past few years. Initially, acid cleaning was employed primarily on a trouble shooting basis for problem jobs. Now, more equipment is being scheduled for acid cleaning on a routine maintenance basis. As this equipment is subjected to repeated exposures of acid cleaning, it becomes imperative that the possibilities for corrosion are understood and that appropriate corrosion control measures are taken. Recently, several cases of unexpected corrosion have been experienced in refinery equipment during acid cleaning. Some of these cases were easy to diagnose while others posed complex solutions. To explore the possibilities for corrosion, a series of laboratory studies have been undertaken. Although this work is not complete, the data are presented here in an attempt to put corrosion during acid cleaning in the proper perspective. As far as possible, attempts have been made to correlate these laboratory results with actual corrosion encountered in refinery equipment.

The acid used for cleaning equipment must be inhibited to prevent corrosion of the metal. At the same time, the inhibitor must not prevent the dissolution of the scale. A wetting agent is needed to counter any oil film that may be present and to permit the intimate contact between acid and scales. Figure 1 shows a typical hookup for an acid cleaning job. Water and steam are mixed together in a header to produce hot water which is subsequently mixed with acid. Samples are collected and titrated to determine the concentration. Both temperature and concentration are controlled by adjusting the proper valve. A return line (not shown) from the equipment is connected to the suction side of the pump, and circulation is established when desired through the solvent input line. This system of heating is preferred over heating coils because the coils would have a high metal skin temperature and would suffer accelerated corrosion.

## Typical Cases of Corrosion During Acid Cleaning

One of the problems frequently encountered is the copper plating of steel parts of the system after cleaning admiralty bundles in heat exchangers. Iron will displace copper ions in solution because of their relative position in the electromotive series. Consequently, any copper dissolved in the system, either from corrosion or solution of corrosion product, tends to plate out when contact is made with steel pumps and



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acid tanks. Generally, this plating is most severe when a circulation-type treatment is employed as opposed to a fill-and-soak method. During circulation any ferrous and cuprous ions can be oxidized to ferric and cupric respectively. These ions can react with the metal in an oxidation-reduction reaction against which most inhibitors are not effective.

## Abstract

Economical, effective and relatively easy to handle, hydrochloric acid is being widely used as a solvent for chemical cleaning of refinery equipment. Inhibitors must be added to retard corrosion during cleaning operations, but these inhibitors must not prevent dissolution of the scale to be removed. Practical temperature ranges for cleaning are given. The corrosion rate increases exponentially with temperature and with acid concentration. The corrosion rates of various metals in 5 percent inhibited hydrochloric acid are given. The influence of inhibitors on the rates of dissolution of iron oxides and sulfides are shown.

Four typical cases of corrosion occurring during acid cleaning are discussed with special safety precautions that should be observed during cleaning operations. The influence of commonly present substances such as hydrogen sulfide is shown to increase the rate of corrosion. 5.9.2

The best preventive is to use the fill-and-soak method, circulating only enough to keep fresh acid in contact with the scales. This is practical since most of the deposits encountered in admiralty equipment are water scales with high solubility in acid. Some advantage has reportedly been gained by grounding the pumping equipment to the system being cleaned.

The good results achieved by grounding may have been accompanied also by reduced circulation. Velocity effects through nozzles, etc., are pronounced when a high concentration of ferric chloride is present. In one case, during acid cleaning of a sweating oven containing steel tubes, a leak developed caused by dirt underneath a manway gasket. By the time the leak was discovered, tightening of the manway cover plates was ineffective. Within a short time the hole had enlarged to the size

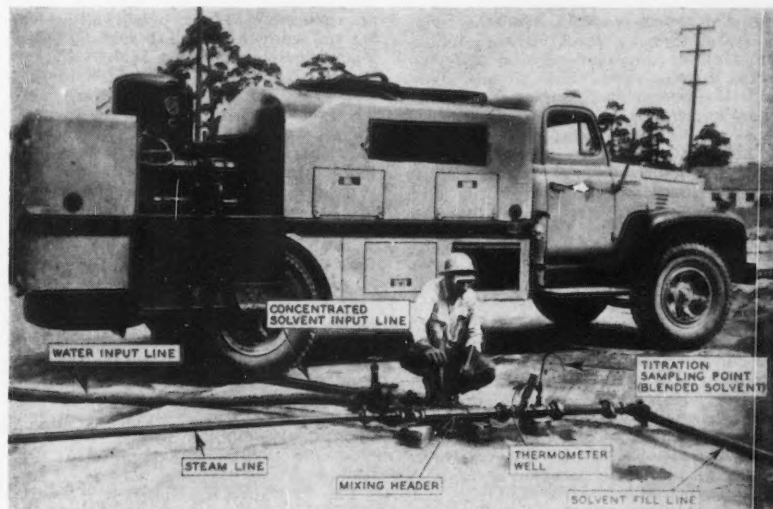


Figure 1—Typical acid cleaning hookup.

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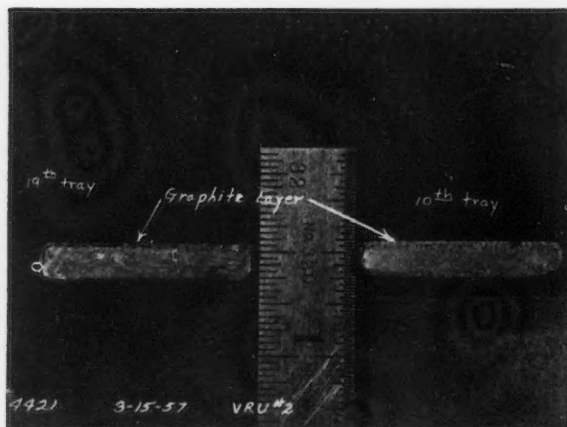


Figure 2—Graphitic corrosion of cast iron caused by excessive temperatures. Note depth of graphitic attack.

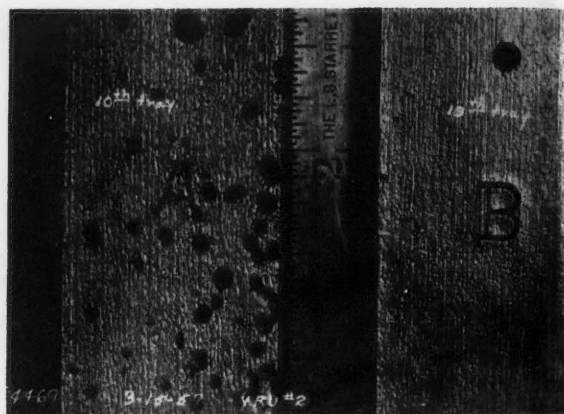


Figure 3—Pitting of carbon steel coupons caused by corrosion product contamination.



Figure 4—Crevice corrosion on plugs from furnace header. The line on the seating surface indicates the depth of the corrosion in the crevice.

of a man's thumb, and the entire contents of the vessel had to be dumped. The actual concentration of ferric iron was not determined, but conditions were known to be favorable to the formation of high ferric iron. Under these conditions the pipe and fittings used to circulate the acid will have a relatively short life.

One of the more severe cases of corrosion occurred when excessive temperatures were attained during acid cleaning of an isobutane tower. In this case, the tower was injected with 115-lb steam in two places to heat the solution and thus to obtain better solubility. Half of the required acid was cascaded to the tower with steam being added. The construction of the tower and the amount of acid were such that most of the acid collected in the bottom of the tower with only a nominal amount on the trays. The steaming was then continued for two hours while more acid was being obtained. During this time temperatures of over 300 F were recorded in the tower. The tower was at a temperature of over 250 F for 2½ hours. The temperatures dropped rapidly when circulation was resumed, but the harm probably was already done. Even during circulation the temperature rose to over 180 F which is higher than recommended although not normally considered serious.

When the tower was opened, severe corrosion was noted on bolts, studs and clips. Corrosion, as is normal, was more severe on the highly stressed areas, such as the sheared edges of clips and bolt threads. Major replacement was necessary in this tower primarily because of

uncontrolled steaming and subsequent excessive temperatures. Heating by injecting steam to equipment must be closely controlled to prevent localized overheating.

On another occasion a tower on a Vapor Recovery Unit was opened prior to a caustic wash and acid treatment. Corrosion test coupons of cast iron and carbon steel were placed on the tenth and nineteenth trays corresponding to roughly 1/3 and 2/3 of the tower height. After acidizing, the strips were removed and examined for deterioration. The cast iron showed evidence of severe graphitization to a depth of .04 inch as shown in Figure 2. The coupon in the lower portion was more severely graphitized than the one nearer the top. Severe pitting to a depth of 0.10 inch was found in the carbon steel coupon exposed in the lower third of the tower with the second strip relatively unaffected. These carbon steel coupons are shown in Figure 3. The corrosion thus appeared to be more severe in the lower portion of the tower. It is significant that the tower was clean at the upper levels with relatively heavy scaling in the lower portion.

The unit was first caustic-washed and steamed to maintain temperature. The tower temperature was reported to be approximately 170 F when filling was started with 7.5 percent acid at 140 F. These temperatures are higher than the 125 F maximum recommended for cast iron. Higher temperatures were used, however, because of a willingness to sacrifice a little corrosion in order to gain increased solubility at the higher temperatures. These higher temperatures are considered largely responsible for the high degree of graphitic corrosion shown on the samples. Examination of the cast iron coupons revealed that they contained a small percentage of combined carbon. Cast irons of this type in general have a greater tendency to undergo graphitic corrosion than the types containing higher proportions of combined carbon. Although the cast iron internals did not show the same degree of graphitic corrosion as the test coupons, the towers do have a history of graphitic attack. A further study of the treating method is warranted.

The fact that the upper steel coupon was relatively unaffected indicates that the acid was well inhibited. The pitted

condition is believed due to the accelerating influences of H<sub>2</sub>S and ferric iron which were generated from the corrosion products in the tower. Deposit analysis from an unexposed bubble cap contained high percentages of Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>, both of which will form ferric iron upon dissolution. Galvanic coupling between other scale deposits, such as FeS<sub>2</sub>, may also have been a contributing factor. Attack was confined to the lower portion of the tower because fouling was limited to this area. Concentration in the tower dropped quickly to 1 percent which indicates the dissolution of large quantities of deposit. The tower was then drained and filled half full with fresh 10 percent acid. Only on the second fill was there a check for ferric iron. This tower was filled from the bottom which means that no check could be made until tower was filled which required three hours. During this period accelerated corrosion caused by the influence of corrosion products, proceeded unchecked in the lower portion of the tower.

Whenever possibilities for ferric iron formation exist, towers and other equipment should be filled from the top or in such a fashion as to facilitate a periodic check for ferric iron. If acid is cascaded over the trays from the top of a tower, spent or contaminated acid can be disposed of continuously at the bottom, thus protecting the tower and internals from corrosion due to corrosion product contamination.

More complex problems have been posed by the etching of furnace headers and plugs found after a catalytic reformer furnace was pickled prior to going on stream. The corrosion on the plugs is shown in Figure 4. Corrosion in the crevice at the seating surfaces was so severe that they leaked on test. Several theories have been advanced to account for this corrosion; among them are preferential corrosion on the highly stressed area presented by the machined surfaces and a galvanic action between the metal and the graphite used in the seating compound. A more probable mechanism, however, appears to be the retention of a low pH chloride solution in the crevice which was not removed during flushing operations. This solution then initiated a concentration cell type action which caused the resulting damage.

What may be a similar type reaction



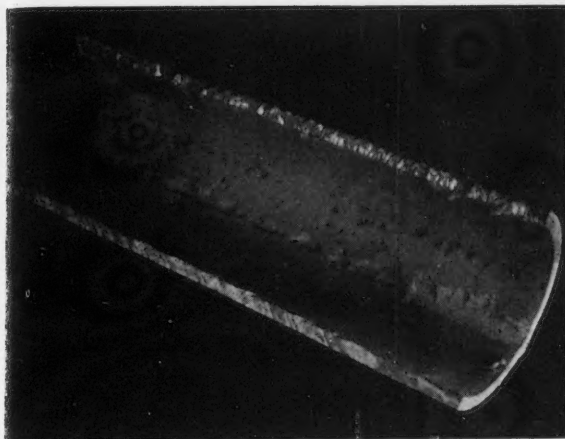


Figure 5—Pitting of furnace tube caused by incomplete flushing.

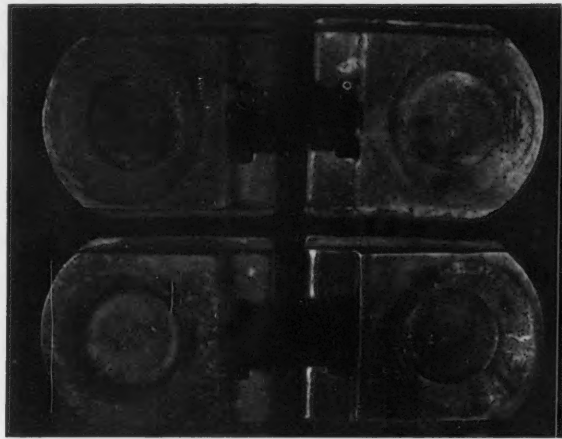


Figure 6—Corroded gates from Type 410 trimmed steel valves after acid cleaning. Corrosion on the sealing surfaces prevents complete shut-off.

occurred in another reformer furnace after repeated acid cleanings for scale removal. Deep scattered pits as shown in Figure 5 were found inside the tubes after a tube failed on hydrostatic test. The unreacted pieces of scale presumably remained in the tube after cleaning which could have retained enough chlorides and acidic constituents to initiate a similar concentration cell mechanism. One suggested answer for this problem would be to flush as adequately as practical and then keep the furnace filled with an alkaline solution until ready to resume operations. Plug crevice corrosion can be avoided by using a plug sealer which would be unaffected by the cleaning solutions.

Another possibility would be to apply a low temperature coating such as a coal tar enamel to the plug which would have to be replaced with a high temperature sealer before the furnace was placed in operation. This problem has not been resolved. Tests in the laboratory have reproduced this crevice effect. Rapid agitation appears to eliminate corrosion perhaps because agitation keeps supplying fresh inhibitor to the surfaces which could become depleted in the crevice. Further research is warranted to overcome this liability of acid cleaning. New and better inhibitors or the application of different cleaning materials may provide answers to the problem.

When stainless steels are present in the system, extreme care must be exercised to insure that the flushing operation is complete. Many inhibitors will give a low corrosion rate on the austenitic steels, but there is no assurance that there will be no chloride ions remaining in crevices in the system. These ions can cause loss of passivity and initiate pitting or they may cause stress corrosion cracking with disastrous results.

Type 410 stainless containing 12 percent chrome appears to be exceptionally susceptible to pitting type attack. This can be explained since Type 410 is on the border line of passivity. The chlorides present can easily shift the passivity creating small anodic areas over the surface of the metal which cause localized cell action and resultant pitting. Pitting can be very pronounced at highly stressed or machined edges. Figure 6 shows gates from Type 410 stainless trimmed steel valves from a solvent extraction unit. Tests had indicated that

TABLE 1—Corrosion Rates in 5 Percent Inhibited Hydrochloric Acid

METAL	Corrosion Rate, Mils Per Hour
Low Carbon Steel.....	0.026
Carbon— $\frac{1}{2}$ Moly Steel.....	0.058
5 Chrome— $\frac{1}{2}$ Moly Steel.....	0.040
Stainless Type 304.....	0.011
Stainless Type 410.....	0.031
Exalloy*.....	0.015
Red Brass.....	0.0012

\* Crane Co. alloy similar to Type 410 stainless.

TABLE 2—Effect of Galvanic Coupling Upon Corrosion in Inhibited Hydrochloric Acid

Galvanic Couple	Corrosion Rate, Mils Per Hour		Potential Measurement
	Coupled	Separate	
Low-Carbon Steel.....	0.036	0.026	Anodic
Stainless Type 304.....	0.0051	0.011	Cathodic
Low-Carbon Steel.....	0.026	0.026	Anodic
Red Brass.....	0.0007	0.0012	Cathodic
5 Chrome— $\frac{1}{2}$ Moly.....	0.035	0.040	Cathodic
Exalloy*.....	0.021	0.015	Anodic

\* Crane Co. alloy similar to Type 410 stainless steel.

the corrosion loss should be small in spite of the recognized tendency towards pitting during acid cleaning. After cleaning, those valves were undamaged which were open during the cleaning cycle. The gates in the closed valves, however, showed severe corrosion in the crevice presented by the gates in contact with the seats. Several of the valves required replacement. All Type 410 stainless should be removed from a system before cleaning if practical.

#### Safety Precautions

Certain recognized safety procedures should be followed on any chemical cleaning job. One important thing to remember is that hydrogen is liberated by the cleaning reaction. Under certain conditions it is possible to confine the hydrogen and set up an explosive mixture in the vessel. Having the vessels properly vented permits the escape of most of the hydrogen from the system as fast as it is formed. The only sure way to eliminate the possibility of an explosive mixture is to maintain an inert gas blanket in the vessel at all times. This normally is not considered practical, however, and effort is directed to-

wards avoiding any action which could possibly ignite an explosive mixture. Smoking, burning or welding around any equipment containing cleaning acid is strictly forbidden.

Many of the deposits encountered in the refinery contain sulfide scales which will liberate large quantities of  $H_2S$ . This volume is particularly large when reformer furnaces are pickled. Refinery workers are aware of the dangers of hydrogen sulfide. Unless provision is made to get rid of the gas, workmen may be overcome by it. Twenty parts per million is the accepted limit for an eight-hour exposure. There are several ways to dispose of this gas, the most common one is to discharge into the blowdown system if the system vents high enough in the air. If no wind exists and large quantities are liberated, however, there is the possibility of the heavier-than-air gas settling around the equipment. Some people prefer to scrub out the gas with a caustic solution. This is done by bubbling the gas through a drum containing caustic. The resultant sodium sulfide must be dumped where it cannot later react to regenerate the hydrogen sulfide. Another possibility is to burn the gas in a furnace to form



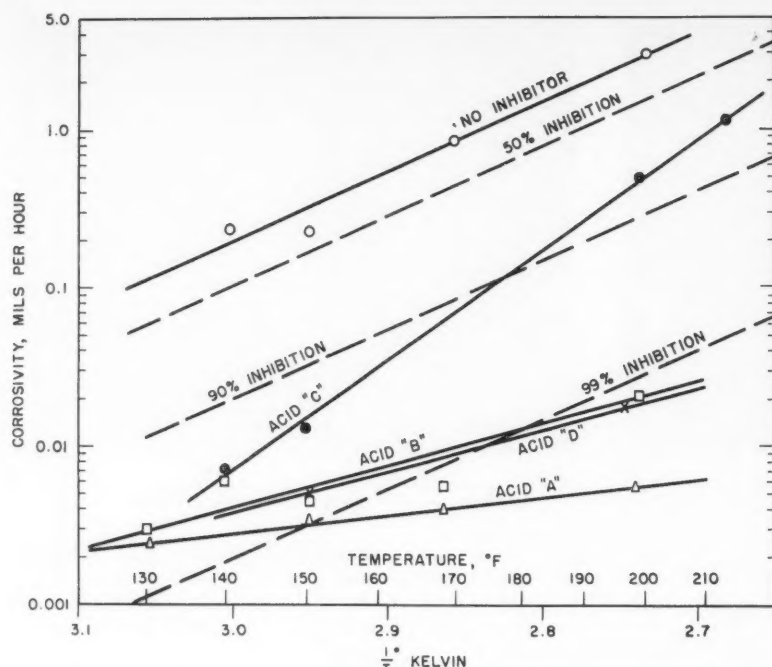


Figure 7—Effect of temperature on corrosion rates of carbon steel in five-percent inhibited and non-inhibited HCl.

TABLE 3—Comparison of Corrosion Rates of Low Carbon Steel and Free Machining Steel in Inhibited Hydrochloric Acids

Temp, Degrees F.	Acid	Acid-Concentration %	Corrosion Rate Mils Per Hour		Acceleration Ratio
			Low C Steel	Free Mach-ine Steel	
150	HCl Non-inhib.	3.75	0.16	5.4	34
		5	0.26	7.0	27
		15	1.1	12.1	11
	Acid D	5	0.005	0.013	2.6
	Acid C	5	0.021	2.24	106
170	Acid B	5	0.005	0.021	4.2
	Acid A	5	0.0035	0.015	2.3
	Acid A	5	0.004	0.023	5.7
	Acid A	5	0.0055	0.055	10
	Acid A	5	0.0055	0.055	10
200	HCl Non-inhib.	5	3	13.8	4.5
	Acid D	5	0.019	0.2	10
	Acid C	5	0.29	10.7	37
	Acid B	5	0.021	3	142
	Acid A	5	0.0055	0.1	18

sulfur dioxide which is toxic but less dangerous than  $H_2S$ .

Other gases may be liberated during the cleaning cycle. Deadly hydrogen cyanide can be liberated in units containing cyanide deposits, e.g., VRU's. A caustic wash is usually employed to remove the salt. Arsine and phosphine are other gases which have been found in vent gases from acid cleaning jobs. Generally, the same precautions for hydrogen sulfide will also take care of the other gases formed. One precaution is particularly important because two men became violently ill when they ignored it. No one should enter any vessel that has been acid treated until that vessel has been properly ventilated to remove any of these gases that may have been trapped there. If common sense procedures are followed, there is little danger of any injury caused by chemical cleaning.

#### Variation in Corrosion Rates Between Alloys

Various metals will react in different ways to inhibited acid. Variations in minor constituents like sulfur and phosphorus can have considerable effect on the corrosion rate. Severely stressed or cold-worked areas, heat affected zones and even grain size may influence the rate of corrosion. For example, Table 1 shows the results of rate determinations of various metals in 5 percent inhibited hydrochloric acid at 150 F for six hours, which would approximate one cleaning cycle. Carbon moly steel corrodes approximately twice as fast as low carbon steel. However, at the rate shown, the total corrosion would be only 0.5 mil per cleaning cycle. The chromium steels also have a higher corrosion rate than the low carbon steels. The high copper alloys have very low corrosion rates primarily because of their relative posi-

tion in the electromotive series. They normally will not corrode by hydrogen evolution in a reducing solution such as hydrochloric acid unless other contaminants are present.

Warning should be given here against the use of hydrochloric acid for cleaning aluminum and other light metals. Most inhibitors are ineffective and cause rapid corrosion. Care should be exercised to assure that no aluminum gaskets are used on equipment to be acid cleaned.

Other complications sometimes arise because of the use of dissimilar metals and the possible galvanic effects when coupled together in such a good electrolyte as hydrochloric acid. Table 2 gives data on the effect of coupling some of the metals in common use. The more anodic metal will corrode faster when coupled with a cathodic material than when metals are placed separately in acid. When coupled, the anodic metal will corrode faster and afford some protection to the other. The rate of corrosion will vary approximately as the relative areas, and the coupling effect becomes significant only when the anodic material is small in relation to the cathode. The time factor, of course, is important. In most cases the treatment time is relatively short. Even if the corrosion rate is high, the amount of corrosion per cleaning is small.

Severe corrosion has been experienced on free machining bolts in equipment being acid cleaned. Free machining steel contains a higher percentage of sulfur than ordinary steel. This sulfur forms iron sulfide at the grain boundaries and promotes chip formation in machining. Table 3 indicates the corrosion rates experienced on low carbon and free machining steels in various cleaning acids at several temperatures. At 150 F free machining steel was corroded 27 times faster than low carbon steel in 5 percent non-inhibited acid. Commercial inhibited 5 percent acids varied in acceleration ratio from 2.3 for acid A to 106 for acid C. At 170 F the acceleration ratio increased to 5.7 for acid A, and at 200 F the ratio was 18. The acceleration ratio for the poorer inhibited acids dropped off at the higher temperatures. This is meaningless because of the high base rate for carbon steel. Only acid A appears to have any utility for free machining steels at 200 F, and this value should be confirmed by further testing.

#### The Effect of Temperature

Temperature plays a dual role during acid cleaning. In general, the higher the temperature the better the solubility of the scale in the acid. The best cleaning job is achieved when every effort is directed toward maintaining a hot solution. As is typical of most chemical reactions, however, the corrosion rate also increases with temperature.

Figure 7 graphically illustrates the effect of temperature on several cleaning acids. In this graph the logarithm of the corrosion rate is plotted versus the reciprocal of the absolute temperature with degrees Fahrenheit also indicated. The straight-line relationship confirms the exponential effect of temperature on the corrosion rate. The selection of a logarithmic scale also permits a greater range of corrosion rates to be plotted on the same graph. At the top of the graph is shown the corrosion rate of 5 percent uninhibited acid. Parallel lines have been drawn showing the rates for 50 percent, 90 percent and 99

percent inhibition. The commercial cleaning acids also at 5 percent concentration have been designated as acids A, B, C and D. Acid A utilizes an inhibitor recently developed for cleaning acid and appears to be less sensitive to temperature than the others. Acid C appears to be poor in this respect. The accepted upper limit has been 170 F for solution temperatures consistent with tolerable corrosion rates. As better inhibitors are developed, higher temperatures can be tolerated. Temperatures should be maintained below 125 F for cast iron. Acid A appears from laboratory tests to have application at temperatures as high as 200 F. However, there may be other factors, and tests at this time are not inclusive enough to warrant this conclusion.

#### The Effect of Concentration

Although the more common usage of acid requires only 5 to 10 percent, some scales (e.g. reformer furnace deposit) may call for 20 to 25 percent acid for best results. Figure 8 shows the effect of concentration on the corrosion rates at 150 F and 200 F respectively. Again the log of the corrosivity is expressed in mils per hour. As might be expected, the corrosion rate increases with concentration. The poorly inhibited acids are more sensitive to concentration changes than the better ones. The better inhibited acids are affected in an exponential fashion but at a lower level. Again acid A appears to be less affected by variations in concentrations. When inhibited acids are diluted, the acid-inhibitor ratio remains constant while the inhibitor concentration in the solution decreases. Since the inhibitor action is conveniently expressed as a plating action on the metal surface, excessive dilution would mean that there is insufficient inhibitor to maintain an intact film.

Most service contractors add enough inhibitor to provide 1 to 2 gallons per thousand gallons of dilute solution. This means that there is proportionately more inhibitor used when dilute solutions are employed. When inhibited acid is purchased, it is imperative that sufficient inhibitor be present to maintain the desired concentration in the solution as it is to be used. Increased concentration will have a greater effect at higher temperatures. This is indicated by the steeper slope of the lines on the 200 F chart.

The function of the inhibitor is to prevent the corrosion of the metal, but at the same time the inhibitor must not seriously retard the dissolution of the scales. Table 4 shows the percentage of inhibition on the dissolution of low carbon steels, iron sulfides and iron oxides by various inhibited 5 percent acids at 150 F. These rates are compared to non-inhibited acid. Although a higher percentage of inhibition is shown for free machining steel than for low carbon steel, the actual corrosion rate is higher because of the much higher corrosion rate of non-inhibited acid on free machining steels. Although Acid C gives the poorest inhibition to attack on steel, it is a better cleaning compound because there is less inhibition on the dissolution of the scale. For example, there is only a 5 percent reduction in dissolving rate for FeS and 13 percent reduction for FeO. In the case of  $Fe_3O_4$  the rate of attack was increased 170 percent probably because of superior detergency.

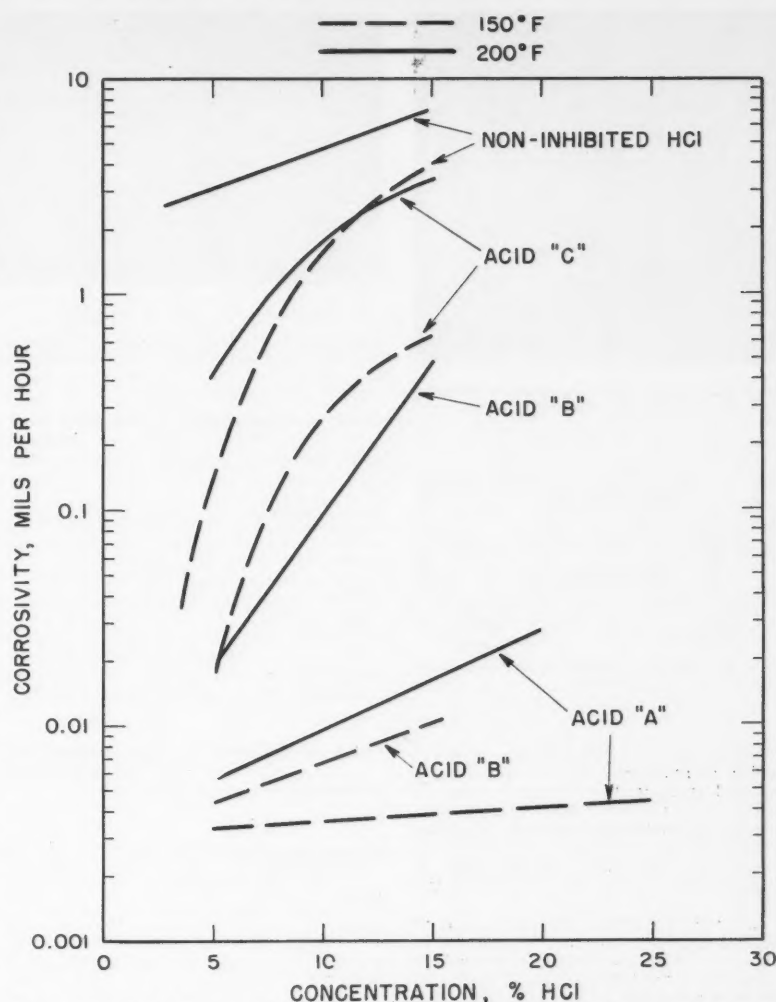


Figure 8—Variation in the corrosivity of acids with increasing concentration at 150 F and 200 F. Broken line represents 150 F; solid line represents 200 F.

TABLE 4—Influence of Inhibitors in Hydrochloric Acid on Rates of Dissolution of Iron Oxides and Sulfides.\*

ACID	% INHIBITION IN DISSOLUTION OF:					
	Low C Steel	Free Machining Steel	FeS	FeS <sub>2</sub>	FeO	Fe <sub>3</sub> O <sub>4</sub>
Acid C.....	66	71	5	(—)	13	—170
Acid B.....	98.2	99.7	50	(—)	38	12
Acid A.....	99	99.9	23	(—)	62	(—)
Acid D.....	98.5	....	15	(—)	75	10

Note: (—) sign indicates slight acceleration of dissolution.

In the case of  $Fe_3O_4$  in Acid C, acceleration of dissolution as compared with plain 5 percent HCl was by 170 percent.

\* All tests at 150 F in 5 percent acid for 2 hours.

The other acids have better inhibition on steel but slow the rate at which the scales will dissolve. This indicates that a knowledge of the scales to be encountered may influence the selection of cleaning acids or at least influence the choice of inhibitor and detergent. A longer contact time may be required for some deposits if the better inhibited acids are used. Most companies engaged in chemical cleaning work provide technical service for deposit analysis and recommendations as to concentration, temperature and time.

#### Influence of Commonly Present Substances

Although ferric chloride will greatly accelerate the corrosion of steel, the question arose as to what other compounds might have a similar effect. Cleaning of refinery equipment differs from water scales and millscale removal in that large quantities of hydrogen sulfide are frequently liberated. Some  $H_2S$  is nearly always present in equipment that has been in contact with hydrocarbons. The rate of corrosion increases when  $H_2S$  is present. Table 5

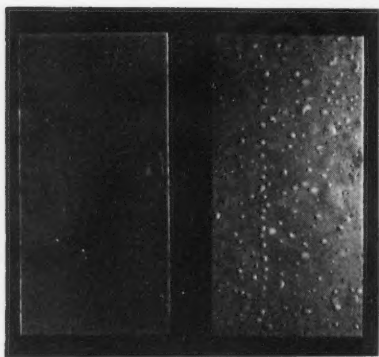


Figure 9—Hydrogen blistering produced in laboratory by introducing  $H_2S$  to cleaning solution.

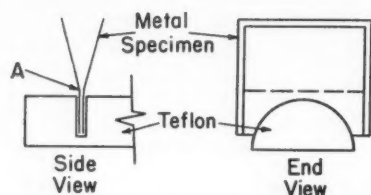


Figure 10—Mounting technique for crevice corrosion studies.

indicates that corrosion by non-inhibited acid is increased 8.5 times when  $H_2S$  is bubbled through the solution. Acids B and C show two to three times the corrosion rate in the presence of  $H_2S$ . The action of the inhibitor in acid D is unique in that the corrosion rate is actually decreased in the presence of the gas.

Another interesting phenomena due to  $H_2S$  in the cleaning acid is shown in Figure 9. The panels were treated in 5 percent inhibited acid for 2 hours at 150 F. The panel on the right had  $H_2S$  bubbled through the solution; the other did not. Note the severe hydrogen blistering on the panel exposed to the  $H_2S$ . This appears to be the same type of action that has been reported in the literature to occur in low pH solutions. The more common type occurs in alkaline solutions in the presence of cyanides. The strong acid here removes the protective sulfide film and permits the atomic hydrogen produced through corrosion to penetrate the clean steel. Hydrogen blistering has not occurred during acid cleaning of refinery equipment, but this test indicates a distinct possibility. Annealing tends to lessen this effect. Some bolt breakage apparently caused by hydrogen embrittlement has been discovered subsequent to acid cleaning operations. Service conditions are also favorable to embrittlement because of the sulfides present. Thus it is not conclusively associated with acid cleaning. Apparently critical stress phenomena are involved here also.

During the acid cleaning of refinery equipment, corrosion products may be encountered which can have a detrimental effect on the corrosion rate. These products may be either water soluble ( $FeCl_2$ ) or water insoluble ( $FeS_2$ ). Table 6 illustrates the effect on the corrosion of steel by the presence of some common substances. These substances may act in two ways: (1) they can release ions into the acid and

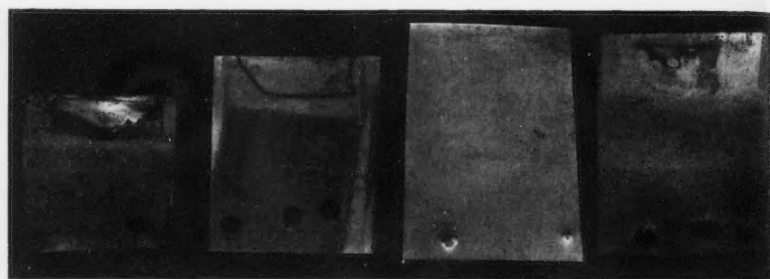


Figure 11—Effect of additives on crevice corrosion of Type 410 stainless steel in cleaning acid. The additives used on the four specimens are: (1) lead acetate, (2) stannous chloride, (3) blank and (4) chloroplatinic acid.

TABLE 5—Acceleration of Corrosion Rate of Steel in Inhibited Hydrochloric Acids by Hydrogen Sulfide\*

Temp., Degrees F	Acid	Ratio = $\frac{\text{Rate in Presence of } H_2S}{\text{Rate in Absence of } H_2S}$
150.....	Plain HCl	8.5
	Acid C	3
	Acid B	2
200.....	Acid C	2
150.....	Acid D	0.8
200.....	Acid D	0.5

\* All tests were run two hours in 5 percent acids.

TABLE 6—Influence of Some Common Corrosion Products on the Corrosion of Steel in an Inhibited Hydrochloric Acid.\*

Corrosion Product Present (% to acid solution)	Corrosion Rate, Mills Per hour	Acceleration Ratio = $\frac{\text{Rate in Presence of Corrosion Product}}{\text{Rate in Absence of Corrosion Product}}$
None.....	.013	
$FeCl_2$ 1%.....	.021	1.6
10%.....	.043	3.3
20%.....	.052	4
$NiCl_2$ 4%.....	.014	1.1
$CrCl_3$ 4%.....	.063	4.9
Powers in poor contact with steel:		
$FeS$ 0.4%.....	.037	3
$FeS_2$ 0.4%.....	.015	1.2
$FeO$ 0.4%.....	.097	7.5
$Fe_2O_3$ 0.4%.....	.038	2.9
Steel embedded in power of $FeS_2$ .....	.63	49
$FeCl_2$ 1%.....	.12	9
20%.....	2.8	220

\* All tests were run with 5 percent Acid C for two hours at 150 F.

thereby increase the rate of attack, or (2) they can form galvanic couples with steel and accelerate the attack by becoming the cathodic component. The chloride salts indicated in Table 6 are all water soluble, and the acceleration effect is therefore caused by the metal ion.  $Fe^{2+}$  and  $Cr^{3+}$  accelerate the attack moderately, but  $Ni^{2+}$  has little influence. These percentages are higher than are usually encountered in acid cleaning, but they do indicate qualitative results.  $FeS$  apparently acts by liberating  $H_2S$  in acid solution and also by release of  $Fe^{2+}$  both of which accelerate the corrosion rate.  $FeS_2$  is not attacked by dilute HCl to any appreciable extent but apparently forms a very active galvanic couple accelerating the corrosion by a factor of 50 when iron is imbedded in  $FeS_2$  powders. In poor contact with steel a small percentage (0.4) had only

a slight effect. The addition of 0.4 percent  $Fe_2O_3$  increased corrosion by a factor of three. This is probably due to the formation of  $Fe^{3+}$  ions when the scale dissolves in the acid. Although not shown,  $Fe_2O_3$  will also result in the formation of  $Fe^{3+}$ .

In this test  $FeO$  accelerated corrosion by 7.5 although the mechanism is not understood. One percent  $FeCl_2$  increased corrosion nine times, and 20 percent  $FeCl_2$  increased corrosion 220 times. The mechanism of corrosion acceleration by ferric ion appears to be a cathode reaction between  $2Fe^{3+}$  and  $Fe^0$  to give 3 ferrous ions, thus bypassing the hydrogen ion discharge reaction. Most inhibitors do not appear to be effective against this type of corrosion. Aware of this fact, most cleaning contractors titrate the solution for ferric iron with approximately 0.4 per-



cent being a maximum allowable. When hydrogen sulfide is present, it probably tends to reduce the ferric iron. Circulation, agitation with air and possible oxidizing agents used in water treatment will tend to oxidize the ferrous iron to ferric and thereby greatly accelerate the corrosion rate.

#### Effect of Crevices on Corrosion

The fact that crevices have a pronounced effect on the corrosion rate has been discussed during pickling of furnace tubes for millscale removal, where the header plugs were corroded. A crevice effect also was encountered on 12 percent chromium trimmed gate valves. These difficulties prompted some test work to explore this phenomena.

Thin strips of Type 410 stainless steel sheet were mounted in a Teflon block as shown in Figure 10, thus creating a crevice between the strips. These blocks, placed in beakers of 5 percent acid containing various additives, were maintained at 140 F for about 17 hours. Upon removal, distinct crevice corrosion was found along the edge where the metal strips were in contact. Typical specimens are shown in Figure 11. No appreciable corrosion was observed away from the crevice area. These tests were duplicated with carbon steel strips and found to be similar. In several cases a thin Teflon gasket placed between the metal strips had no effect upon the crevice corrosion, indicating that metal-to-metal contact is not required. This type of corrosion is probably caused by depletion of the inhibitor in the restricted crevices where diffusion occurs slowly, followed by the establishment of highly active and localized corrosion cells on the metal surface.

Many additives were tried to minimize this crevice effect. Stannous chloride, lead nitrate and lead acetate appeared to be the most effective in reducing the crevice effect. Specimen 2 in Figure 11 had stannous chloride added to the solution. Specimen 1 had lead acetate, Specimen 3 is a blank and Specimen 4 shows the accelerating effects of chloroplatinic acid. These tests were then carried one step farther;  $H_2S$  was added to the system since it is present in nearly all refinery streams. The  $H_2S$  cancelled the beneficial effects

of the tin and lead salts in reducing the crevice corrosion. While the addition of the salts might be beneficial in acid cleaning of new units, the salts probably would not help in other units where sulfidic scale is present. The sulfides probably act by precipitating the lead and tin as the respective sulfides.

This appears to be one of the major problems of acid cleaning refinery equipment. Many similar jobs have been performed with no apparent ill effects. Rapid agitation seems to eliminate this attack. Better inhibitors may be developed to combat this effect, or additives may be found which will not be affected by sulfides in the stream. Additional investigation must be performed before equipment of this type can be cleaned with complete safety.

#### Conclusions

1. A good cleaning acid must be inhibited to protect the equipment, but at the same time the inhibitor must not prevent dissolution of the scale.
2. The corrosion rate increases exponentially with temperature. The maximum allowable temperature will vary depending upon the inhibitor. Around 170 F is a generally 'accepted' maximum for carbon steel equipment; 125 F is the accepted limit for cast iron.
3. The corrosion rate increases with concentration but is sometimes compensated by using increased quantities of inhibitors.
4. The presence of  $H_2S$  increases the rate of corrosion two to three times. Hydrogen blisters were also produced in laboratory tests.
5. Ferrous and chromic ions increase the corrosion rate moderately, depending upon the concentration.
6. Ferric ions greatly accelerate corrosion by cleaning solutions. A one-percent solution increased the corrosion rate nine times. The 'accepted' limit for ferric iron is 0.4 percent by weight in the solution.
7. Free machining steels were found to corrode 3 to 100 times as fast as low carbon steel depending upon the inhibitor and the temperature.
8. Severe pitting has been encountered on 12 percent chromium trim in steel

gate valves. It is recommended that all 12 percent chromium be removed from equipment before acid cleaning.

9. A crevice effect is sometimes encountered during pickling of furnace tubes around the header plugs. Additives have been found effective for new work but hydrogen sulfide, if present, nullifies the beneficial effects.

10. Hydrochloric acid is an effective cleaning tool and can be safely used under most circumstances if the proper procedures are followed. Disregard for these procedures, however, may result in severe corrosion of refinery equipment.

#### Acknowledgment

The authors wish to acknowledge the work of Dr. A. J. Freedman and Bernard Grushkin in evaluating the crevice attack phenomena and the corrosion of various alloys. G. Van Hees and R. Betris assisted in the laboratory tests. The authors are also indebted to R. J. Hafsten for his encouragement and assistance in the preparation of this paper.

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Any discussions of this article not published above  
will appear in the June, 1959 issue

# A Polarographic Study of the Corrosion Of Iron and Some Ferrous Alloys by Sulfur Dioxide\*

By F. J. BOWEN<sup>(1)</sup> and A. H. GROPP<sup>(2)</sup>

## Introduction

SULFUR DIOXIDE is one of the most serious offenders of the gases which may contaminate an atmosphere, because of its corrosive effect upon many metals when moisture is present. Thompson has stated that sulfur dioxide is the predominate corroding agent in atmospheric corrosion.<sup>1</sup> Clark has shown that this gas is the chief constituent of the air which affects indoor corrosion and that sulfur dioxide readily attacks steel and nickel when present in an atmosphere of more than 70 percent relative humidity.<sup>2</sup> Tolley has also shown that in the presence of water vapor the corrosive effect of sulfur dioxide is accelerated.<sup>3</sup> When sulfur dioxide is present in aqueous solutions of either sulfuric or hydrochloric acid, the corrosion rate of iron in the medium is proportional to the sulfur dioxide concentration.<sup>4</sup> Work by Binger, Wagner and Brown indicates that in high concentrations of sulfur dioxide in aqueous solution the corrosion rate of mild steel is excessive.<sup>5</sup>

## Corrosion of Iron and Steel by Sulfur Dioxide

A study of the manner in which sulfur dioxide attacks iron and ferrous alloys should prove interesting, and useful information might be obtained by following the changes in sulfur dioxide content of a solution\* containing an iron specimen.

## Polarographic Corrosion Studies

The use of the polarograph in corrosion studies is neither new nor common. R. M. Burns first used this instrument for the study of corrosion in 1937 when he compared the rates of solution of two grades of lead in distilled water.<sup>6</sup> This was done by measuring changes in the lead ion concentration in solution. Van Rysselberghe and his co-workers used the polarograph to study the effects of oxygen and carbon dioxide in the corrosion of metals.<sup>7,8,9</sup> This work was done by measuring changes in the concentration of various corrosive agents as corrosion proceeded. Since that time, a number of investigators have used a polarographic method for corrosion studies. For the most part, changes in the concentration of the corroding agent have been determined.

The polarograph permits a qualitative and quantitative analysis for the constituents of a solution by means of measured reduction currents resulting from the application of gradually increasing cathodic potentials to a dropping mercury or rotating platinum electrode immersed in a solution containing reducible ions of molecules. A cathodic wave is recorded by any of several means, the abscissa being the applied

electromotive force and the ordinate being the current passing through the cell. The height of the wave thus recorded is a measure of the limiting diffusion current of the ion or molecule being reduced, and therefore of the concentration of the substance. The reduction potential of the substance, which is the point at which the wave is recorded, is characteristic of that ion or molecule. In general, substances having reduction potentials of from 0.0 volts to -2.4 volts with respect to the saturated calomel cell may be determined in this manner. The quantity of a substance reduced in the polarographic analysis is so small as compared to the bulk concentration of the same material that any changes in concentration caused by the analysis may be disregarded in the ranges that the instrument may be used. Thus, successive analyses of the same solution may be made at intervals as rapidly as the mechanical factors of the instrument permit. The time required for an analysis varies from ten seconds to five minutes depending upon the instrument and the manner in which the analysis is performed. Kolthoff and Lingane state that the accuracy of the polarographic method is  $\pm 2$  percent within the concentration range  $10^{-2}$  and  $10^{-4}$  molar, and  $\pm 5$  percent between  $10^{-4}$  and  $10^{-5}$  molar.<sup>10</sup> This is equal to that of other micro analytical methods.

There are a number of limitations to the use of the polarograph in corrosion work, but where applicable, this method permits relatively rapid and controlled study of a process.

Studies made by polarographic analysis of the solutions do not define the method of the attack, i.e., general surface attack, pitting, or other forms of corrosion. The major objection is that the solution studied must be a good electrolyte, or if not, must have an ionizable salt added in excess to form a good electrolyte. This necessity often rules out the use of the instrument, particularly in studies concerning the effect of distilled water or non-conducting organic liquids in corrosion.

Gosman and his co-workers first showed the possibility of reducing sulfur dioxide at the dropping mercury electrode and obtained well defined waves in one-normal hydrochloric acid.<sup>11</sup> They also showed that no reduction occurred in neutral or alkaline solutions. Kolthoff and Miller extended this work and showed that, while sulfur dioxide is readily reduced in acid solution, the wave height found is dependent upon the pH of the solution and that with constant concentration of the sulfite or sulfur dioxide the wave height decreased with increasing pH.<sup>12</sup> In strongly acid solution only one wave was found, but at a pH of six, two small waves appeared. No reduction occurred at a pH greater than six. The half-wave potential in one-normal hydrochloric acid was found to be -0.2 volts with respect to the saturated calomel elec-

## Abstract

A study was made of the manner in which sulfur dioxide attacks iron and ferrous alloys. A polarographic method was used to follow the changes in the sulfur dioxide concentration. Tests were made on one pure iron and seven low alloy steels exposed to sulfur dioxide over 12-hour periods. Extensive data are reported on the rate of consumption of sulfur dioxide by the various test samples. A corrosion mechanism involving the reduction of sulfuric acid by iron to give the sulfide is discussed. 4.3.2

trode although this varied with the supporting electrolyte.

## Apparatus

A Sargent-Heyrovsky Model XII photographically recording polarograph was used for all work described herein. A dropping mercury electrode was used in all cases. A shunt ratio of 50 was used, corresponding to 0.2450 microamperes per millimeter of wave height.

The cell used is shown in Figure 1. The essential difference between this cell and those commonly used in polarographic work is the addition of a glass barrier at the bottom of the cell to restrict the anode pool and to allow the metal samples to be placed in such a position that corrosion products would not contaminate the large mercury anode. When allowed to come in con-

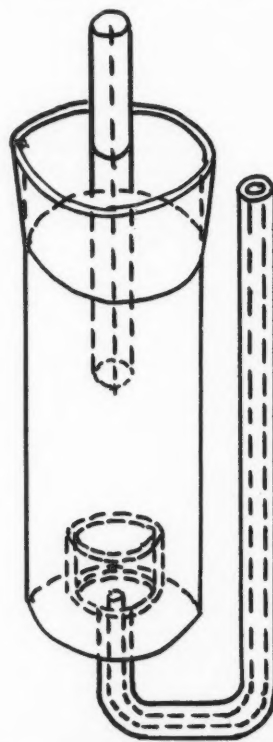


Figure 1—Polarographic cell, illustrating anode barrier.

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\* No distinction will be made in this paper between sulfur dioxide in solution and/or sulfuric acid ( $H_2SO_3$ ).

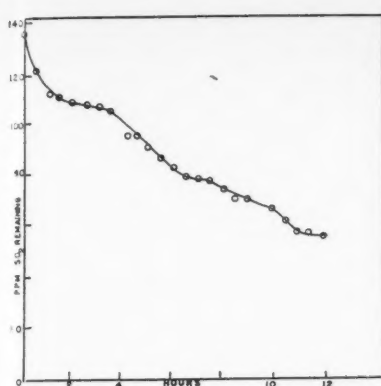
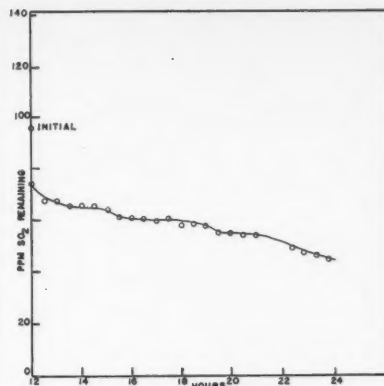
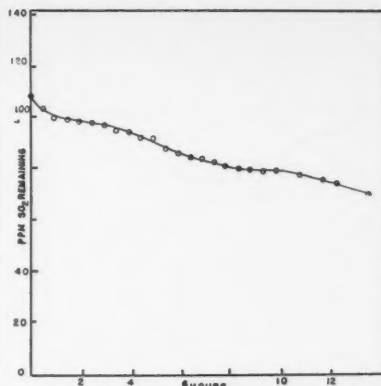
Figure 2—Consumption of SO<sub>2</sub> by Sample 1 during first 12-hour period.Figure 3—Consumption of SO<sub>2</sub> by Sample 1 during second 12-hour period.Figure 4—Consumption of SO<sub>2</sub> by Sample 2 during first 12-hour period.

TABLE 1—Composition of Iron and Ferrous Alloys\*

Sample No.	COMPOSITION, PERCENT							
	C	Mn	P	S	Si	Cu	Ni	Cr
1	0.02	0.04	0.006	0.020	0.002	0.007	0.006	0.001
2	.04	.31	.008	.018	.011	.012	.01	.003
3	.03	.47	.07	.06	.003	.27	.03	.03
4	.04	.49	.07	.042	.003	.05	.01	.10
5	.09	.38	.11	.026	.72	.33	.03	.92
6	.13	.42	.010	.020	.21	.16	2.1	.13
7	.04	.46	.014	.027	.004	.06	3.5	.16
8	.10	.40	.014	.024	.32	.028	.28	5.9

\* Obtained from the Research Laboratory, United States Steel Corporation, Vandergrift, Pennsylvania

tact with the anode, these products caused such a variation in potential that measurements of the diffusion current and the half-wave potential could not be made. Placed between the glass barrier and the outer wall, the metal samples rested on edge at the bottom of the cell. No perceptible change in the wave height of sulfur dioxide could be found when the contents of the cell were stirred by gentle shaking. This indicated that the barrier did not affect diffusion significantly.

The use of other cells was tried without success. An "H" cell with the metal sample in one arm and the anode in the other was considered a suitable apparatus, but this was found to be unsatisfactory because homogeneity of the solution could not be obtained after the corrosion process had begun.

Because a loss of sulfur dioxide occurred when rubber stoppers were used, all connections were made with ground glass fittings. A glass tube was used between the mercury reservoir and the capillary within the cell. A stopcock in the tube permitted the flow of mercury to be interrupted as desired.

Two cells were used, both having a volume of 12 milliliters. Both cells were calibrated for loss of sulfur dioxide either by loss from solution or by use of the cell components. If the ground glass stopper was properly inserted into the cell, no measurable loss of sulfur dioxide occurred for over 100 hours.

The first cell adopted for use was calibrated for wave height of the sulfur dioxide for known sulfur dioxide concentrations by analyzing the contents of the cell and duplicate portions of the solution with which the cell had been filled. The official method of the American Public Health Association was used for this analysis.<sup>12</sup> The second cell was

calibrated against the first by determining the wave heights for aliquot portions of the same sulfur dioxide solution in the two cells. This was necessary since wave height is dependent upon various factors which are in turn dependent upon the capillary used. Calibration curves for the two cells were then constructed.

A concentration range of between 15 and 150 parts per million of sulfur dioxide could be covered adequately. Although measurements of wave height for concentrations below 15 ppm could be made, this low concentration range was not advisable. The precision of the chemical analysis used to determine the calibration curve was not good at low sulfur dioxide concentrations although it was possible polarographically to detect differences in SO<sub>2</sub> content as small as two parts per million at the instrument sensitivities employed.

#### Materials and Procedure

Metal samples used in this work were obtained from Dr. R. B. Mears of the United States Steel Corporation and Dr. I. A. Denison of the National Bureau of Standards. With the exception of the Sample 1, all samples were low alloy steels similar to those which have been used in corrosion tests by the two organizations. The compositions of the various materials are shown in Table 1.

The sulfur dioxide solutions used as corrosive media were made by bubbling tank SO<sub>2</sub> into 0.1 N potassium chloride (AR quality). A large volume of strong stock sulfur dioxide solution was maintained under a nitrogen atmosphere at all times. The exclusion of oxygen was necessary to prevent the reaction between oxygen and sulfur dioxide which would cause a decrease in sulfur dioxide

concentration. Aliquot portions of the stock were diluted with oxygen-free\* potassium chloride solution to obtain the desired reagent concentrations for this work. Although solutions which contained equal quantities of SO<sub>2</sub> were difficult to obtain by this procedure over periods of time, the procedure was preferable to that of obtaining SO<sub>2</sub> in solution by mixing calculated quantities of sulfites or bisulfites with acid buffered solutions. This latter procedure has been used by other investigators.<sup>12</sup>

Early experiments showed that, when gaseous SO<sub>2</sub> was bubbled into the supporting electrolyte, an immediately measurable reduction wave was obtained which did not change with time (no steel specimen present). However, if solutions of sodium sulfite or sodium bisulfite were introduced into a buffered acid supporting electrolyte to produce sulfur dioxide (or sulfurous acid), the height of the polarographic wave increased for some time, except in very acid buffers when a maximum height was reached immediately. This point is worthy of further investigation and may account for statements that sulfur dioxide must be measured in strongly acid solution and that the wave height is dependent upon the pH.<sup>10</sup> Since a feasible method of measuring the wave height of sulfur dioxide, which was relatively independent of pH in the desired ranges, was obtained by the use of gaseous SO<sub>2</sub>, the point was not investigated further in this work.

However, to clarify whether changes in pH within the cell would significantly affect the wave height recorded for a given SO<sub>2</sub> concentration within the pH ranges studied, the wave heights were recorded of equal quantities of SO<sub>2</sub> in unbuffered one-tenth molar potassium chloride solutions and in buffered citrate solutions. Between a pH of 2.00 and 4.00 the pH of the solution did not significantly affect the recorded wave height. For example, a wave height of 42 mm was recorded for solutions containing 62 ppm of SO<sub>2</sub> whether the solution was unbuffered potassium chloride at pH 3.15 or the buffered citrate at pH 3.80. This relationship was determined in steps of 0.2 pH units between pH 2.00 and pH 4. This range was adequate to include all of the measurements taken in this investigation.

Specimens 1 cm wide by 2 cm long

\* The term oxygen-free is used in the sense that oxygen could not be detected polarographically and was thus less than 10<sup>-6</sup> to 10<sup>-7</sup> molar in concentration.



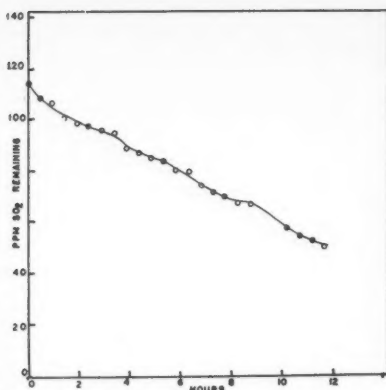


Figure 5—Consumption of  $\text{SO}_2$  by Sample 3 during first 12-hour period.

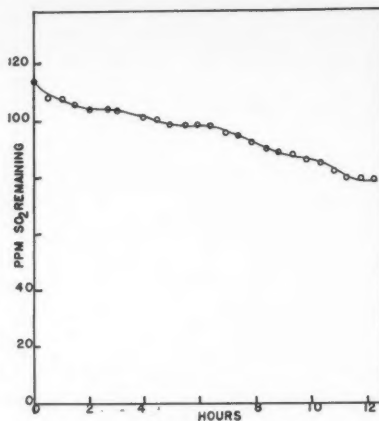


Figure 6—Consumption of  $\text{SO}_2$  by Sample 4 during first 12-hour period.

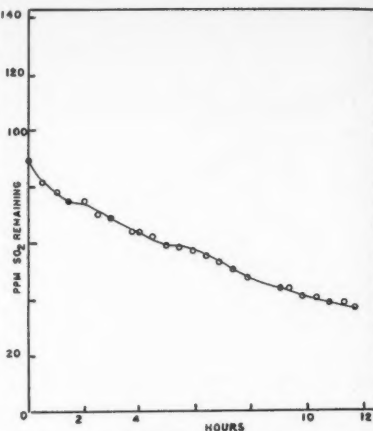


Figure 7—Consumption of  $\text{SO}_2$  by Sample 5 during first 12-hour period.

were used. The gauge varied, but in all cases the total apparent surface area was between 4.35 and 4.50 square centimeters. The specimens were annealed for one-half hour at 950 C. (1740 F) and then furnace cooled. The primary purpose of this treatment was to remove strains and cold work caused by shearing the specimen to size because the ratio of the surface area of the edges to the total surface area was relatively large. Before use, scale was removed by wire brushing, and each specimen was polished with successive grades of emery cloth through 3/0. The specimen was then thoroughly washed with 95 percent alcohol and finally with distilled water to obtain a water break-free surface. The surface was dried by blotting with filter paper.

The specimen was placed in position in the test cell which was continuously flushed with nitrogen until the solution to be used was introduced. The cell was filled to overflowing with the sulfur dioxide containing solution and the ground glass stopper inserted. Care was taken that no air space existed between the solution and the stopper.

The first measurement of  $\text{SO}_2$  concentration was made immediately. Successive measurements usually were made at intervals of one-half hour although shorter periods were sometimes used when it became apparent that the  $\text{SO}_2$  concentration was changing rapidly. The mercury flow was interrupted between measurements. When measurements between 12 and 24 hours were desired, the initial measurement was made and the test cell permitted to remain undisturbed for twelve hours. The wave height of the  $\text{SO}_2$  was then recorded as usual at half-hour intervals.

Measurements of wave height were made at the half-wave potential in all cases, using standard techniques. These measurements were made to the nearest one-half millimeter which corresponded to one part per million of sulfur dioxide at the sensitivity at which the apparatus was used.

### Results

The results obtained by plotting the sulfur dioxide concentration against time are shown in Figures 2 through 11. Each figure represents the measurements made with one specimen only for a 12-hour period. The measurements that were made during the period between 12 and 24 hours are shown for two samples only (Figures 3 and 11). The shape of the curves during the second 12-hour period was the same as the first twelve

hours although in most cases the overall rate of sulfur dioxide consumption decreased.

Figures 2 and 3 show the consumption of sulfur dioxide by pure iron (Sample 1). The initial rate of consumption is high and persists for almost two hours, after which the rate of reaction decreases and is extremely low for a two hour period. At the end of this time, there is a sharp increase in the consumption of the sulfur dioxide, again for two hours, and followed by another period of inactivity. This lowered activity lasts only a short time before the rate again increases, this time for about three hours after which the rate decreases. Figure 3 shows that the general picture of the process from 12 to 24 hours is much the same although the over-all rate is greatly decreased.

Figure 4 shows the consumption of sulfur dioxide by Sample 2, a low carbon steel of low alloy content. Again, the initial two hour period shows the greatest rate of consumption of the corroding agent. This is followed by decreased activity for two hours and then an increased rate of consumption. This increased rate continues for more than four hours when a second inactive period occurs. After approximately two hours, the rate again increases. However, there is almost continuous consumption of sulfur dioxide for the entire 12-hour period although the rate is much less than that for pure iron.

The consumption of sulfur dioxide by Sample 3, which is also of low alloy content, is shown in Figure 5. This sample contained a relatively large percentage of copper. The greatest rate of consumption of sulfur dioxide is in the first two-hour period, and this is also followed by a lower rate for the next two hours. After this, the sulfur dioxide is consumed at the same rate for successive two-hour periods up to 12 hours although the curve shows short periods of decreased activity at regular intervals.

Figure 6 illustrates the reaction rate for Sample 4 which is slightly higher in alloy content than Sample 3 although of the same general composition. Sample 4 contains the highest percentage of manganese of the samples tested. The curve obtained for this steel is similar to that obtained for Sample 3 with periods of activity and inactivity. As before, the greatest rate of consumption of sulfur dioxide occurs in the first two hours, followed by a period of inhibition. The rate for the first 12 hours

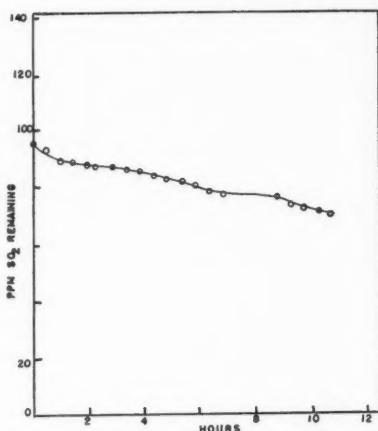


Figure 8—Consumption of  $\text{SO}_2$  by Sample 6 during first 12-hour period.

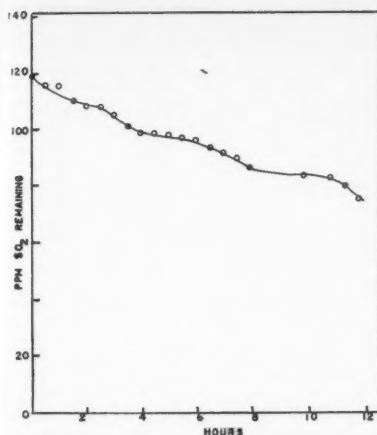
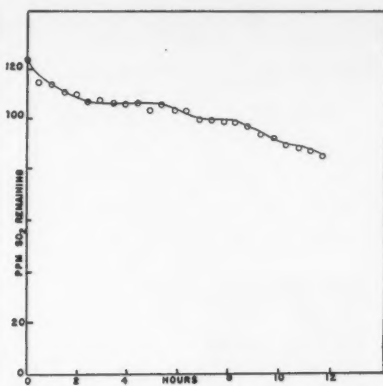
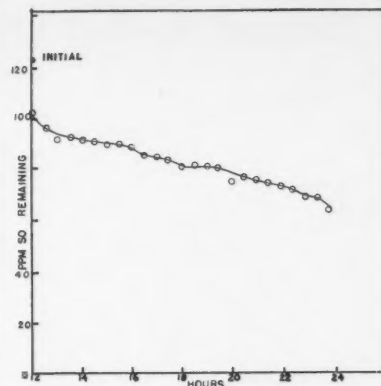
appears to be divided roughly at approximately six hours. The rate for the last six hours is slightly greater than for the first six since the first period of inactivity lasts for almost four hours before activity increases.

Figure 7 is for Sample 5. This sample was the highest in phosphorous, silicon and copper of any of the alloys tested. An inactive period is not shown until after the sixth hour although the  $\text{SO}_2$  consumption rate is considerably less after the first two hours. The inactive period is about an hour. The consumption of sulfur dioxide is then resumed without another inactive period although the rate varies and is not constant.

Sample 6 is shown in Figure 8. This sample contains 2.1 percent of nickel. Again in the first two hours the consumption of sulfur dioxide proceeds at the greatest rate. After this, a period of inactivity occurs for two hours, followed by a two-hour period of increased consumption. This is followed by three hours of decreased action, and then again, increased activity on the part of the sample.

Figure 9 shows the results for Sample 7 which contains 3.5 percent of nickel. In the first 12 hours, the periods of reaction and passivity are well defined compared to the previous sample. Four periods of activity and three periods of inhibited action are to be observed.

Sample 8, containing 5.9 percent of

Figure 9—Consumption of SO<sub>2</sub> by Sample 7 during first 12-hour period.Figure 10—Consumption of SO<sub>2</sub> by Sample 8 during first 12-hour period.Figure 11—Consumption of SO<sub>2</sub> by Sample 8 during second 12-hour period.

chromium, is shown in Figures 10 and 11. This type of steel may be known as 4-6 Chromium alloy. The first two hours show a rapid consumption of sulfur dioxide and are followed by a three-hour period in which there is apparently no use of the corrodant. A two-hour period of activity is then followed by two hours of complete inactivity. Increased activity is again resumed at the eighth hour and continues for the rest of the 12-hour period. The last 12 hours assume an almost steady rate of sulfur dioxide consumption with two short periods of inactivity between the sixteenth and the twentieth hours.

Table 2 summarizes the information obtained for the first 12 hours for each sample. While the general corrosion picture is depicted, that of periods of activity followed by periods of inhibition, reference to the curve for a particular sample gives a truer picture of the corrosion process. In all cases, the greatest consumption of sulfur dioxide by the sample occurred during the first two hours.

Although curves for only one specimen of each alloy for a given period are shown in each figure, the shapes of the curves are reproducible. This is shown in Figure 12 in which the reaction curves for three different specimens of the same alloy in three different solutions are shown.

The logarithm of the remaining sulfur dioxide concentration versus the time is plotted for three samples in Figure 13. If the logarithm of the concentration of a component of a reaction is plotted against time during the period in which the reaction is taking place, a straight line will result if the reaction is of first order with respect to that particular component. The corrosion process, therefore, is of first order with respect to the sulfur dioxide for at least the greater portion of the first 12-hour period. Only plots for Samples 2, 3 and 8 are shown. Two of these represent the extreme limits of the rates found; the third represents a sample rate falling between these limits. All others studied gave the same type of plot, the slope of the line being the only major difference.

#### Discussion

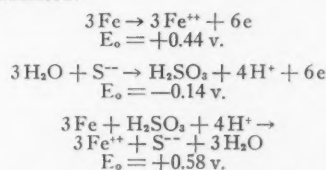
When the cells were cleaned at the end of a corrosion period, the odor of hydrogen sulfide was present. When samples of metal were placed in a

stronger sulfur dioxide solution than those which were used for corrosion tests, the odor of hydrogen sulfide became very strong in a short time. Apparently, sulfur dioxide is reduced either wholly or in part to the sulfide ion. Simple qualitative tests indicated that the black coating was ferrous sulfide.

Although the solutions used were acid and had a pH of less than 4.0, no measurable quantity of hydrogen was evolved during the corrosion process.

Any mechanism for the corrosion which may be postulated must therefore account for the presence of the sulfide and the lack of evolution of hydrogen gas in the acid solution.

The most probable mechanism is that of the reduction of the sulfurous acid by the iron to give the sulfide. Using oxidation potentials from Latimer, the following reactions and values may be calculated:<sup>14</sup>



By the use of the equation  $\Delta F_o = -nFE_o$ , it may be calculated that  $\Delta F_o$  for the above reaction is -80.2 kilocalories. This is large enough for the reaction to be readily possible.

This reaction would account for the presence of the sulfide which was found and the black coating of ferrous sulfide on the metal sample. The initial rapid consumption of sulfur dioxide would be caused by the reduction of the sulfur dioxide and the consequent formation of a ferrous sulfide coating upon the surface of the sample. As the coating upon

the metal becomes thicker and more stable, no more sulfide dioxide can reach the metal. This would account for the period of apparent inactivity following a period of sulfur dioxide consumption.

The equilibrium constants for hydrogen sulfide are given by Latimer as  $1.1 \times 10^{-7}$  and  $1.0 \times 10^{-14}$  respectively.<sup>14</sup> By combination of  $K_1$  and  $K_2$ , the following expression is obtained:

$$\frac{C_{\text{H}^+} \times C_{\text{S}^{--}}}{C_{\text{H}_2\text{S}}} = 1.1 \times 10^{-21}$$

Latimer also gives the solubility product for FeS as  $4.0 \times 10^{-19}$ , so that the sulfide ion concentration must have the value  $4 \times 10^{-19}/C_{\text{Fe}^{++}}$ . If this is substituted into the equilibrium expression for hydrogen sulfide, the equation becomes:

$$\frac{C_{\text{H}^+} \times 4 \times 10^{-19}}{C_{\text{H}_2\text{S}} \times C_{\text{Fe}^{++}}} = 1.1 \times 10^{-21}$$

and, by rearrangement, the concentration of the ferrous ion must have the value:

$$\frac{C_{\text{Fe}^{++}}}{C_{\text{H}_2\text{S}}} = \frac{C_{\text{H}^+} \times 3.6 \times 10^3}{C_{\text{H}_2\text{S}}}$$

The maximum sulfide ion concentration that could be obtained in this work is in the neighborhood of  $2 \times 10^{-8}$  M if all of the available sulfur dioxide were converted to the sulfide. The maximum pH that was studied was approximately pH 4. These would be the conditions for the minimum solubility of FeS. By substituting these values into the expression  $\text{Fe}^{++}$  concentration above, the  $\text{Fe}^{++}$  is calculated to be approximately  $1.8 \times 10^{-2}$  M. Even under minimum solubility conditions for these experiments, a large proportion of the ferrous sulfide formed could be dissolved by the solution.

Thus, the ferrous sulfide coating upon the iron sample is dissolved in the acid media. If the rate of dissolution is low,

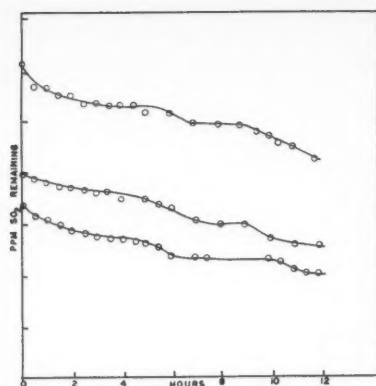


Figure 12—Consumption of  $\text{SO}_2$  by Sample 8 showing reproducibility of measurements.

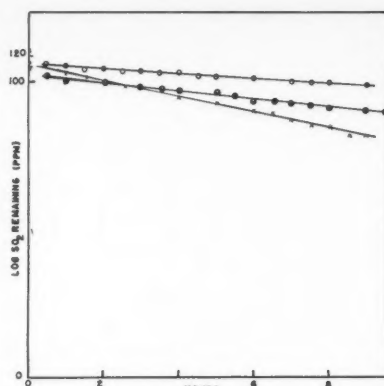


Figure 13—Logarithmic plot of consumption of  $\text{SO}_2$  by Samples 2, 3 and 8; X = Sample 2; ● = Sample 3; ○ = Sample 8.

some time will elapse until the remaining coating becomes permeable and sulfur dioxide can again react with the metal. When this happens and the consumption of the sulfur dioxide is resumed, a new period of increased activity is shown. The process becomes cyclic in nature and may continue to remain so. During the initial stages of the attack, the periods of inactivity will be relatively short because of the greater solubility of the ferrous sulfide during this time. If the concentration of the sulfur dioxide is limited as in these experiments, it is possible that inactivity periods will become longer as the attack continues and the solubility of ferrous sulfide decreases. Eventually, a steady condition might result if enough ferrous sulfide remained upon the surface of the metal that the attack proceeded at the same rate as the dissolution of the coating.

The formation of the original ferrous sulfide coating must depend upon the relative rates of several reactions and at least two equilibria. The first of these is the reaction of the ferrous ion and the sulfide ion to form ferrous sulfide and the equilibrium concerned with the dissolution of the ferrous sulfide. The other reaction and equilibrium are those concerned with the hydrogen ion and the sulfide ion in the formation of hydrogen sulfide. It must be realized that the situation at hand is not analogous to the precipitation of ferrous sulfide by hydrogen sulfide in acid solution as in ordinary qualitative analysis. In that case, the sulfide ion must be formed by the ionization of hydrogen sulfide, and this is prevented by the hydrogen ion already present. In the present case, the sulfide ion is formed by the reaction at the surface of the iron sample. At the interface between the solution and the metal surface, a high local concentration of ferrous ion and sulfide ion exists, and the hydrogen ion concentration is diminished because of the reduction that takes place. The rate of reaction between the ferrous ion and the sulfide ion must be more rapid than that establishing the equilibrium between the diminished concentration of the hydrogen ion and the sulfide ion. The formation of ferrous sulfide results. The probability of this reaction must be considered since the ions are present in an interface between the solution and the solid metal sample and not in the bulk of the solution where the hydrogen ion-hydrogen

sulfide equilibrium would be most effective.

Although a mechanism for the corrosion of ordinary iron and steel alloys has been postulated which satisfactorily explains the data obtained, there is always the possibility that some other reaction, although less apparent, may actually take place. It is also possible that the corrosion process may involve several reactions operating concurrently. The reduction of a solution of sulfur dioxide may yield many possible products. The two best known are thiosulfate and free sulfur. No free sulfur was observed at any time in a corrosion cell. The half-wave potential of the thiosulfate ion is given by Kolthoff and Lingane as  $-0.15$  v. with respect to the saturated calomel electrode.<sup>10</sup> Since this is within the range covered in the measurements of the sulfur dioxide concentration, the presence of any thiosulfate ion should have been apparent in the polarograms which were made. This was not the case, and no evidence was obtained to indicate the presence of this ion. It may be noted here that there is no recorded half-wave potential for a sulfur compound above that of sulfur dioxide in acid solution. Since no wave other than that of sulfur dioxide was observed between 0.00 volts and approximately  $-1.4$  volts, it would seem that any sulfur compound that can be further reduced polarographically is not indicated. Since free sulfur would not be indicated polarographically in the present study, the possibility should not be overlooked that it may have been present as a transitory state in the reduction process or in quantities so small as to escape visual detection.

The products of corrosion by sulfur compounds may be complex and consist of polysulfides as well as the simple sulfide. This must be considered since the values for the free energy of formation of the sulfide ion and the various polysulfide ions differ by relatively small amounts. Latimer calculates the free energies as follows:  $\text{S}^{2-}$ , 22,100 cal.;  $\text{S}_2^{2-}$ , 21,800 cal.;  $\text{S}_3^{2-}$ , 21,100 cal.; and  $\text{S}_4^{2-}$ , 19,400 cal.<sup>11</sup>

### Conclusions

Table 3 lists the percentage of the total sulfur dioxide consumed by each of the eight samples in the two 12-hour periods. Samples 1, 3 and 5 show the most activity and Sample 4 the least.

TABLE 3—Sulfur Dioxide Consumption in 12-Hour Periods

Sample No.	Percent Sulfur Dioxide Consumed	
	0-12 Hours	12-24 Hours
1.....	59	31
2.....	32	32
3.....	56	39
4.....	31	21
5.....	60	29
6.....	30	42
7.....	39	24
8.....	32	33

A comparison of Tables 1 and 3 indicates the following:

Samples 1 and 2 show the beneficial results of the addition of manganese. If this is coupled with the addition of copper, as in Sample 3, all the benefits of the manganese are lost. A decrease in the amount of copper and a small increase in chromium produces the most resistant alloy in the series as is shown by the behavior of Sample 4.

A small decrease in manganese and large increases in copper and chromium, Sample 5, eliminates all benefits accrued by the addition of manganese and chromium. This sample also contains the highest amounts of phosphorus and silicon.

Samples 6 and 7, which have the highest nickel content, and Sample 8, which has the highest chromium content, also show definite improvement.

Therefore, manganese was the best inhibitor of the corrosion, and chromium and nickel also lessened the attack of sulfur dioxide. In direct contrast, copper seems to be the most detrimental of the additives. Silicon might also be placed in this class.

### Acknowledgment

Acknowledgment is given to Dr. R. B. Mears and Dr. I. A. Denison for supplying the metal samples to be used in this work as well as for suggestions concerning the work.

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# Cathodic Protection and High Resistivity Soil—A Sequel\*

By H. C. VAN NOUHUYS

## Introduction

**M**OST CORROSION ENGINEERS are agreed on the wisdom and economy of placing coated pipe lines under continuous, or solid cathodic protection. They find, however, that solid protection of bare pipe lines normally is not economically feasible because of its larger current requirements. Hence, engineers resort either to spot protection based on careful survey, or reconditioning and coating of the old pipe after which solid protection is added at relatively low cost.

Most of the published experience to date concerns spot protection of bare pipe in low resistivity soils.

The author in an earlier paper<sup>1</sup> described the design, installation, and economic aspects of a solid protection system consisting of 25 rectifiers along 350 miles of 8-inch pipe, from Port St. Joe, Florida to Atlanta, Georgia. About 325 miles, or 93 percent of this pipe was bare.

Figure 1 is a map of the pipe line, the alternate black and white spaces representing the protective spreads from each of the 25 rectifiers. For the sake of simplification only the even numbered rectifiers are listed.

Today, some five years later, there is evidence that solid protection of bare pipe lines in very high and ultra-high resistivity soils is both feasible and economically justified provided over-protection is avoided by use of a modified remote electrode criterion design.

Table 1 shows what is meant by the term "high resistivity soil." The average resistivity at pipe depth probably is close to 100,000 ohm-centimeters and is classed as "very high."

The sizing and placement of rectifiers for this system was based on lowering the pipe potential a minimum of 0.3 volt with respect to a copper sulfate electrode placed at remote earth 300 feet from the pipe.<sup>2</sup> Reasons for using this design were:

- Supplementary tests using two copper sulfate electrodes at anodic points indicated that anodic areas were suppressed wherever 0.3 volt or more lowering of pipe potentials was being obtained. This test is made by placing one electrode over the pipe and the other 5 feet out at right angles. Henceforth, this arrangement will be referred to as "the 2 electrode test."

- This design called for the least number of rectifiers, lowest current density, and consequently the least cost of any other criteria.

Since in the final analysis, time and leak record prove the adequacy of any cathodic protection system, it might be well to review the leak record shown in Figure 2. The line was constructed in 1941, the first leak occurred in August 1943, and another in July 1945. By late

## About the Author



HERBERT C. VAN NOUHUYS, until recently corrosion and research engineer for Southeastern Pipe Line Company, Atlanta, Ga., has formed his own independent corporation to design and engineer cathodic protection systems for transmission, distribution, and industrial applications. He has been engaged in various engineering phases of pipe line construction, operation, and maintenance since 1939 and has devoted full time to corrosion mitigation for the last 12 years. After receiving a BSE degree in electrical engineering from the University of Michigan in 1934, he spent several years as a test, applications, and research engineer in the heating, air conditioning, and refrigeration fields. During five years of WW II he held assignments with the Signal Corps and Air Corps as a staff Supply and Property Officer, and Technical Officer at Ground Radar Research and Development Labs, respectively. He remains active as a major in the Air Force Reserve, has authored several technical papers, is a former member of AIEE, ASH&VE, and Engineering Society of Detroit. His NACE record includes four years election to three posts and appointive service, under the last four NACE presidents, as chairman of the Regional Management Committee and Director ex officio.

1946, 14 rectifiers were installed at corrosive areas to give spot protection until October 1952, at which time the design and installation of the 25 rectifier remote electrode system was completed. The leak total was seven in lieu of an estimated 70 with no protection, as indicated by the well known and widely accepted logarithmic leak frequency curve for long lines. Ten corrosion leaks have occurred in the five year period from October 1952 to October 1957, whereas the estimated no protection curve indicates that by this time total leaks would have numbered 370.

Although each leak was analyzed to determine its cause, only enough time will be taken here to describe a typical leak in each of the four categories established.

## Rectifier Downtime

### Category A Leaks

Leaks which would not have occurred had the rectifiers been kept on the line at specified output will be referred to as Category A leaks. None of this pipe received protection for approximately 6 years and only portions of it received protection during a 12-year period. For this reason the piping was on the verge of leaking at many locations. All that was needed to cause a leak in some cases was for a rectifier to go down for

## Abstract

Additional information is given to supplement an earlier paper dealing with the installation and economic aspects of a 25 rectifier system used to provide solid protection along 350 miles of bare 8-inch pipe in ultra-high resistivity soil. Four basic types of leaks are discussed.

Analysis of the leak record is made from data obtained at each leak location in an effort to properly allocate those leaks due to inadequacy of the criterion as opposed to leaks resulting from rectifier down-time or other unusual causes. A total of 10 leaks which occurred during the first five years of operation are considered.

A summary is given of the savings to be expected from use of this method of design over other criteria as well as precautions to be taken in its use. 4.5.3

a day or a week, or for an unduly high line pressure surge to occur. As necessary rectifier and ground bed maintenance is speeded up and as frequency of reporting rectifier operating data is maintained at once per week, it is believed leaks from this source will become rare.

An example of a Category A leak occurred January 14, 1957, 1.64 miles south of a rectifier normally operating at 50-54 amps output and 19½ miles north of a motor-generator set with a normal output of 40-46 amps. Needless to say the nearer unit furnished practically 100 percent of the protection at that point. This rectifier was installed in October, 1946, giving this "leak to be" a 5 year and 10 month start and a pit estimated at 95 percent of perforation depth before any protection was applied.

After protection was applied the average output for 7 years from 1946 to 1953 was only 10 amps. This output was enough, however, to give a pipe to soil potential difference of 0.55 volt (1.01 on, 0.46 off) to a remote electrode and 0.10 volt (0.62 on, 0.52 off) to a local electrode and thus either halt or slow down corrosion activity. A 30 day down time in 1953 and 60 day down time in 1954, however, in spite of 27 and 21 ampere average outputs allowed corrosion to proceed and reduce wall thickness to its absolute safe minimum. In 1955, with a 47 ampere average and only 4½ hours down time, further penetration was not experienced. This did not prove true in 1956 where the output averaged 45½ amperes and where there were three days down in August and 10 days at the end of December. On January 14 a slight surge resulted in a breakthrough. Pipe to soil potentials 4 feet from the leak collar were as follows:

## P/S(Local) P/S(Remote)

Rectifier On	.705	1.55
Rectifier Off	.520	.46

Difference .185V 1.09V which is over three times the minimum requirement of 0.3 volt. A 2-electrode test gave the following data:

W	E
—251 mv	On —210 mv
—14 mv	Off — 8 mv
—237 mv	Diff —202 mv

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Figure 1—Cathodic protection system for Southeastern Pipe Line Co.

Prior excavation at the leak necessitated taking 2-electrode data four feet from the leak collar. The low minus values ( $-14$  mv and  $-8$  mv) with the rectifier off indicates that although cathodic at this point, the pipe could easily have been appreciably anodic four feet away, whereas the high minus values ( $-210$  mv and  $-250$  mv) with the rectifier on indicate that even with abnormal attenuation these should be sufficient to suppress corrosion right at the leak.

#### Category B Leaks

Category B leaks are defined here as leaks which occurred in areas either known to be less than the minimum pipe to soil potential necessary for protection or found by test at time of leak to be below standard. There are three or four known areas still below standard due to excessive currents being picked up by foreign lines and returned via bond wires at the crossings. All of the current in a bond represents a total loss to the protected line. Many of these bonds are being eliminated by coating the line from 400 to 1000 feet at the crossings. In such cases there is remarkable improvement in the foreign line potentials. Figure 3 shows an example of this which is believed self-explanatory even though results are somewhat amazing. A Category B leak occurred on April 23, 1956, in a  $\frac{3}{4}$  mile long area which was below the 0.3 volt criterion due to allowing a new foreign line to have 8 amperes in a

bond 3.85 miles away. This supposition is believed correct because the leak occurred less than three months after the bond was made and tests showed that the closed bond caused the P/S potential in the leak area to change 0.2 volt with the rectifier on and off, whereas with the bond open, the change became 0.3 volt. In this particular case the 8 ampere bond was later eliminated when the foreign line installed its own rectifier system.

Many 2-electrode tests made in conjunction with remote P/S tests at anodic points indicate that better than 9 times out of 10, suppression is accomplished where a 0.3 volt difference is obtained. Inasmuch as the rule is not infallible it is necessary when checking out a newly designed system to use the 2-electrode test in conjunction with the P/S potential test at anodic points nearest the mid-areas or electrical centers between rectifiers. If these weakest areas are receiving adequate current to effect suppression, chances are that going either way toward a rectifier will show still better results. This, however, is not always true; two leaks have occurred close to rectifiers where the remote criterion was more than satisfied, yet the 2-electrode test indicated lack of suppression. These leaks might have been avoided if 2-electrode tests had always been made along with the P/S tests when checking out the system. Experience has shown that when an

TABLE 1—Soil Resistivity Classification

RANGE IN OHM-CM	CLASS
10-1,000	Very Low
1,000-5,000	Low
5,000-10,000	Medium
10,000-25,000	High
25,000-100,000	Very High
100,000-1,000,000	Ultra-High
1,000,000-Infinity	Super-High

anodic spot is found by the 2-electrode test, the rectifier should be tapped up sufficiently to cause the millivoltmeter pointer to swing at least as far in the cathodic direction from zero as it was in the anodic direction with the rectifier off.

#### Category C Leaks

Category C will refer here to those leaks which occurred due to pine or other types of roots or objects causing cells or shielding which no practical amount of cathodic protection could have prevented. One large pine tree area was inspected and although many roots were along the pipe, the pipe was in excellent condition. It appears there will always be some unusual circumstance to arise and cause a leak, which circumstance would be so unusual that it would be uneconomical to attempt to eliminate the possibility of its recurrence.

A Category C leak occurred February 13, 1956, only 0.78 mile from a rectifier which had had no down time since installation in November 1951 except for two weeks in October-November, 1954. Pipe to soil data were as follows:

	P/S (Local)	P/S (Remote)
On	1.22	3.2
Off	.38	.7
Difference	.84V	2.5V

It is noted that these potentials are high enough to more than satisfy any criterion. The crew had noticed roots growing on the pipe when clamping the leak, and tests at one of these small  $\frac{1}{4}$  inch diameter roots with a millivoltmeter indicated the pipe to be 16 mv positive to the root. This indicates that even a 0.85 volt local electrode criterion is not always enough to stop certain types of concentration cells. Just as Sir Humphrey Davy knew that "a very feeble chemical action would be destroyed by a very feeble electrical force," so it appears that occasionally on a bare steel pipe line chemical cell action requires above standard electrical force to insure its destruction.

#### Category D Leaks

All leaks not contained in Categories A, B, & C are considered due to inadequacy of the remote electrode criterion when used without benefit of the additional 2-electrode test. An example of this type of leak (referred to here as a Category D leak) is as follows. On April 8, 1957, a small leak was found 10.91 miles north of one rectifier and 11.86 miles south of the other. Pipe to soil data were as follows:

	P/S (Local)	P/S (Remote)
On	.310	1.35
Off	.225	.56
Diff. =	.085V	.79V

The 0.79 volt lowering of potential to the remote electrode is about  $2\frac{3}{4}$  times the 0.3 V minimum specification.

A 2-electrode test could not be made directly at the leak clamp due to the pipe having already been raised out of contact with the soil for cleaning. However, it was made 150 feet south with the following result:

W.	S/S	E
-147mv	Both On	-150mv
-60mv	Both Off	-75mv
87	Diff.	75

When these tests can be made directly at the leak or within 2-3 feet, they invariably show anodic (+) readings with both rectifiers off. Both rectifiers had an excellent record of operation. Records on down time were begun four years previously, with only 50 hours and 6 hours down time, both in November 1956. It is believed a leak from this down time would have shown up within 30 to 60 days.

Whenever leaks are believed due to inadequacy of the criterion (i.e., Category D leaks), the adjacent rectifier or rectifiers are tapped up to give greater current output which is equivalent to approaching the next higher criterion. In another paper it was stated: "Our experience has shown that where both the remote electrode criterion and the 2-electrode test are satisfied, protection is adequate."

#### Reduction In Leaks

Of the 10 leaks mentioned, three were in Category A, one in Category B, three in Category C, and three in Category D. This means then, that by giving top priority to rectifier and ground bed maintenance so that units are never allowed to run below specified output, and answering patrol plane outage reports at once, a 30 percent reduction or three out of every ten leaks might be eliminated. Practically, of course, down time cannot be completely eliminated. It means also that by eliminating the three areas known to be below standard and any newly discovered areas, by coating and wrapping and consequent elimination of bonds or at least reduction of bond current, a 10 percent reduction in leaks can be attained. However, as far as the 3 or 30 percent, Category C leaks are concerned, it is obvious that it would be far cheaper to let these super-concentration cells "burn through" than to attempt to supply enough cathodic protection to the bare pipe to stop them. And finally it means that if when a Category D leak due to inadequacy of the remote criterion is discovered, the rectifier output is raised to approach or meet the requirements of the next higher criterion so that no more leaks occur in this area for this cause, then eventually another 30 percent reduction in leaks can be attained. It is believed then, considering all four types of leaks, that during some future five year period the leak frequency may be reduced to one per year in lieu of the two per year record for the first five year period.

Having reviewed the leak record and analyzed the leaks, it is then necessary to determine whether or not this solid protection system is economically justified.

The original paper<sup>1</sup> explained the physical need for such a system based on a mounting leak record, several hundred bell hole inspections, and a long

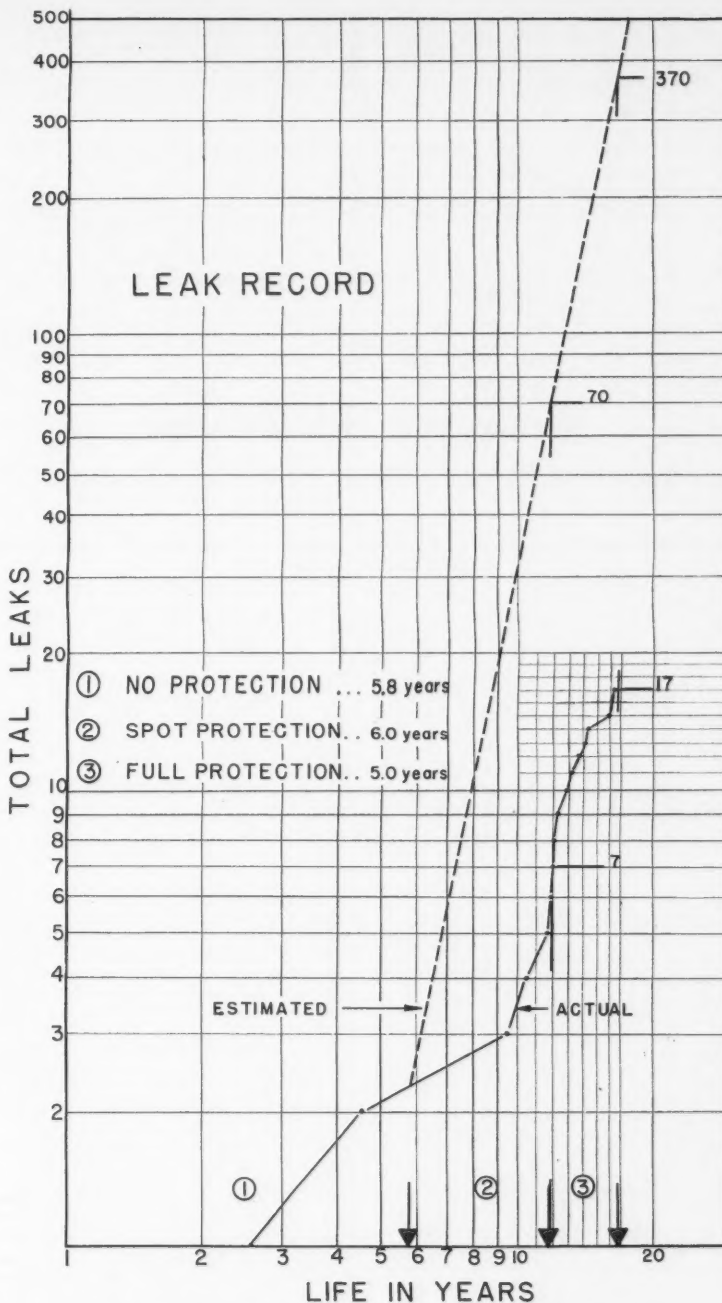


Figure 2—Leak record with no protection, spot protection and full protection.

lines survey which revealed 835 anodic areas. The average cost per year for these five years including power, maintenance, and depreciation is \$43,000. The average cost per leak is \$1,500. At this rate it is obvious that if the system prevented only 30 leaks per year from occurring, savings would amount to \$45,000 which would more than pay for the operation of the system. Following the leak frequency curve of Figure 2, the number of leaks prevented was

$$\frac{(370-70)}{5} = 60 \text{ per year average—a value of } \$90,000, \text{ and a net saving of } \$47,000 \text{ per annum. Those who are fa-}$$

miliar with the various methods of plotting estimated leaks without cathodic protection will appreciate the conservative method used.

Comparison of the cost estimate made five years ago with the actual results is believed to be of interest:

#### Power

Power was estimated at \$21,486 per year and turned out to be \$20,116 or 6½ percent less, due partly to increased average efficiency as a result of replacing seven single phase units with three phase, and partly to the fact that average DC output of the system over the



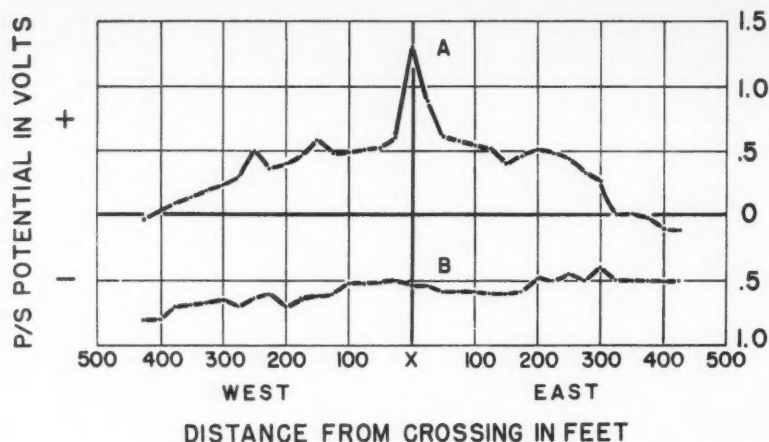


Figure 3—Pipe to soil potentials over 10-inch coated line without bond. Curve A represents reading before taping 800'-8" bare line under protection; Curve B represents reading after taping.

TABLE 2—Estimated Comparison of Protection Criteria for 350 Miles 8" Bare Pipe in High Resistivity Soil

No.	Criterion	Units Req'd	% Now Protected	Av. Density Ma/Sq. Ft.	Cost \$/Mi./Yr.
1	Remote P/S Pot. Diff. = .3V.....	25	110	.20	123
2	Remote-2 Elec. P/S Pot. Diff. = .3V..	25	100	.22	129
3	McCollum ECM .....	25	100	.22	129
4	Remote P/S Pot. = .85V.....	40	62.5	.35	197
5	Local P/S Pot. Diff. = .1V.....	50	50	.44	246
6	Local P/S Pot. Diff. = .3V.....	150	16.6	1.32	738
7	Local P/S Pot. = .85V.....	175	14.3	1.54	861

period was only 94 percent of the minimum desired output.

#### Maintenance

Maintenance was originally estimated, or rather underestimated at \$100/unit/year for a total of \$2,500 and turned out to be \$684/unit/year for a total of \$17,098. At the time the engineer made this estimate, he was not aware of the extent to which:

(a) Termites attack neoprene jacketed-rubber insulated ground bed cables, nor the extent to which,

(b) Certain ground bed resistances would rise during drought periods, necessitating replacement with higher voltage rectifiers or adding rectifiers in series to obtain necessary voltage capacity to insure adequate current output, nor the fact that,

(c) Lightning would prove too much for pellet type protectors used on rectifier DC outputs, necessitating their replacement with the Thyrite Type, nor the extent to which,

(d) Farmers in the vicinity of ground beds stapled their fencing to trees. This allowed trees to pick up current which proceeded to take the easy low resistance route along the fence to the pipe crossing. The current then severed the fence wires as it jumped into a cathodic tree trunk, thence off of anodic roots to the pipe. For this reason pipeliners either insulate the fence from the trees or substitute good dry fence posts.

#### Depreciation

These few lessons from the school of experience are already proving valuable

in holding down the cost of maintenance and improving system operation.

Depreciation was originally \$5,625 per year, but actually ended at \$6,075 due to adding \$9,000 to capital investment in 1955 for 3-3 phase rectifiers, 3 motor-generator sets and 19 air patrol signals.

This sequel would not be complete without a discussion of the estimated costs of protection if other commonly used criteria had been used. Table 2 is an estimate, listing various design criteria in ascending order of number of rectifiers required, current density produced and cost per mile.

Number one in Table 2 is the remote electrode criterion alone used to lower pipe to soil potential 0.3 volt. As explained previously it was found that this criterion was not adequate in all cases. If 25 units give an estimated 90 percent protection when operated at an average current density of 0.20 ma/sq ft, then if operated at 0.22 ma/sq ft, its requirements are exceeded by 10 percent in so far as this criterion is concerned. However, by augmenting this criterion with the 2-electrode tests as a check, it is believed that 100 percent protection can be attained with the same number of installations as before, but operating at 10 percent increased output. Actually some units will remain at the same output while occasionally tests may require that one be raised as much as 50 percent in order to suppress certain areas. Criterion 2 then is very close to the present situation. The McCollum Earth Current Meter is placed in position 3 with the same number of units and current density as No. 2, as a result of tests made at anodic spots midway

between all rectifiers. The ECM checked the combined remote plus 2-electrode specifications, but of course is a more time consuming method which few engineers use today.

The remaining criteria represent increasing overprotection in high resistivity soils. The commonly used "over the pipe" local electrode, 0.3 volt difference, requiring 175 units and producing a current density of 1.32 ma/sq ft at a cost of \$738 per mile would amount to \$215,000 excessive cost per year, or \$1,075,000 for the five year period and would have prevented only three leaks due to inadequacy of the remote criterion and one due to a below par area.

The 1953 paper mentioned that a 60 percent complete, similarly designed rectifier system was being installed on the remaining 110 miles of 6 inch bare pipe from Atlanta to Chattanooga. This system was completed in November 1953, and its five year period was in November of this year. The average current density, 0.42 milliamperes per sq ft, is twice that of the 350 miles, 8 inch line and its 13 rectifiers protect an average of 8.3 miles each as compared to a 14 mile average on the 8 inch system. Soil resistivities going north from Atlanta are apparently only 50-60 percent as high as those from Atlanta southward to the Gulf.

Remote electrode design, it is interesting to note, automatically takes soil resistivities into account in spacing of rectifiers. Seven leaks have occurred in the 50 months since system completion, of which three are in Category A, two in Category B, and two in Category D. Although cost per mile of this system is expected to be greater due to lying in lower resistivity soils, economics of this design will be easily justified when final leak record analysis and comparison with other criteria is made.

Although corrosion engineers in the low resistivity soil areas have found little or no use as yet for the remote placement of the copper-sulfate half-cell, it is admirably suited for use in the ultra-high resistivity soils in automatically taking into account the large IR drop in the soil. This large IR drop occurring between the bare pipe and 300 feet of high resistance soil to remote earth is comparable to the large IR drop (pipe to soil potential) occurring between a thinly but well-coated pipe and only two or three feet of soil when measured in conventional manner to an electrode over the pipe. The coating represents practically all of the drop over a 3/32 inch thickness. On bare pipe the ultra-high resistivity soil may be thought of as being a very imperfect coating many feet in thickness, but nevertheless effective in producing low attenuation characteristics when cathodic protection is applied and measurement is made to include the full IR drop across the thick coating.

When testing coated pipe in high resistivity soil it is found that differences in potentials with rectifier "On" and "Off" are only slightly greater with the remote electrode than with the local electrode. This is because the soil drop is only a small percentage of the total drop. On bare pipe these differences are much greater because IR drop through the soil becomes a high percentage of the total drop.

The school of thought which advocates the elimination of all soil IR drop by taking measurement at the pipe surface to obtain true pipe potential surely

realizes that without soil drop, pipe potential would be zero. If IR drop helps to put potential at the pipe surface, the question arises as to why all of it should not be included in measurements.

If as is believed, current density required for protection lessens as soil resistivity increases, then a potential criterion is needed that will automatically detect and correct for the particular soil being tested. This is what the remote 2-electrode criterion does. Testing and design on this basis will result in less overprotection than the local electrode criterion, yet the two-electrode portion of the test will, with rare exception, assure adequate potential to suppress any local anodic spot encountered.

### Summary

In summary it can be said that:

(1) Although cathodic protection is one of the most desirable investments a pipe line can possibly make, many thousands of dollars are no doubt wasted annually in over-design due to the fact that excessive protection is not easily apparent.

(2) The five year test experience with this system has proven to the author the advisability and excellent economics of approaching the question of "minimum current requirements" from the low side rather than the high side.

(3) Although a spot protection system for bare pipe will give excellent return on investment, it is believed that in most cases a solid protection system based on the remote electrode—2 electrode criterion design will produce an even more favorable return.

(4) Engineers are cautioned against use of other criteria in high resistivity soils as representing overprotection, excessive cost, and increasing problems of electrolysis damage to foreign structures.

(5) Engineers having plant located in the lower resistivity soils are invited to test this dual criterion (as, for example, between two existing rectifiers to determine first hand if suppression can be accomplished with less power.)

### Acknowledgment

To the cooperation and experience of many fellow NACE members is due the success of this system.

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### DISCUSSIONS

Questions by T. J. Maitland, American Telephone and Telegraph Co., New York, N. Y.

1. Please explain in more detail the remote—2 electrode method of measuring protection criteria.

2. How can effect of high negative soil potentials to structures parallel to the pipeline within a mile of the ground bed be reduced?

3. Mention was made by the speaker that bonding between the pipeline and foreign structures will be required to reduce interference effects. Does the author agree with this and is his company agreeable to supplying the current necessary to nullify the effect of the interfering current on the foreign structure?

Replies by H. C. Van Nieuhuys:

1. The procedure followed in using the combination remote-2 electrode criterion was as follows,

(a) Place interrupters of different time cycles in adjacent rectifiers and proceed to location of the anodic spot nearest the electrical center. By electrical center is meant the point of intersection of the respective pipe to soil potential difference curves. At this point each rectifier generates the same p/s potential difference which should not be less than 0.15 volts to a remote electrode. Assuming we find that with both units on we have 0.3 volt or more p/s potential than with both units off, we are then ready for,

(b) A two-electrode test previously described (see references 1 and 2). If the location does not test anodic with both units off, the next nearest location should be tried. Usually an anodic test can be found best adjacent to a swamp, creek crossing, or road ditch. Assuming anodic readings average 20 millivolts with both units off, at least 20 millivolts cathodic indication should be obtained with both units on, otherwise rectifier tap-up is warranted.

As was previously mentioned, if the 0.3 volt difference criterion is satisfied, the second test also is usually satisfied,

but since this is not always true the 2-electrode test should be made when checking out the system. The remote electrode test may be said to insure economy of design while the 2-electrode test is the watchdog insuring against overly intensive local calls breaking through the defensive system.

2. Such pickup potentials, so named because they indicate current pickup, have effectively been reduced on farm fences lying in close proximity to ground beds, by inserting a strain insulator in each strand of barbed wire at intervals of 100 feet-300 feet as required by the particular installation. It is suggested that installation of insulation joints in other structures such as communications cables, pipe lines, railway tracks, etc., would be similarly effective.

3. During the last three years we have found by actual tests similar to that described in Figure 3, that bonds to a bare line under cathodic protection can be successfully eliminated by coating the bare line for an adequate distance. Bare lines with their high current requirements can ill afford heavy foreign line drains, since every ampere in the bond is a total loss to the line under protection. We have had three leaks attributable to allowing bond currents to foreign lines which had unknowingly reduced our potentials below their safe minimum. Although the practice of bonding is an old one, the author is of the opinion that where possible, independence of structures is preferable for the following reasons,

- (a) By eliminating bonds, interstructure resistance is raised, foreign lines pick up less current, and hence discharge less.
- (b) Any serious tendency of a foreign structure to discharge can be largely eliminated by coating the line under protection at the crossing, and adding cathodic protection to the former inasmuch as cathodic protection of all coated structure is now recognized as good policy and is fast becoming standard practice.
- (c) Foreign structures have in the past suffered leaks due to open bonds caused by lightning. Independent systems would eliminate many hours of calculations, tests, meetings, bond adjustments, and headaches for the foreign line engineer whose potentials are always varying due to bond current variations over which he has little or no control.
- (d) From the standpoint of the protected line engineer, careful survey must be made each time he approves a new bond, to determine the extent of loss of protection. As new lines continue to increase, this policy results in a creeping paralysis which saps the strength of the protected line requiring tapup or even addition of new rectifiers. In cases where a line is receiving approximately the same amount as it is giving at other points, all is well. But where the situation is one-sided and the currents are of appreciable magnitude such as was the case of one foreign structure which had ten bonds for a total of 20 amperes, management felt justified in requesting remuneration in view of a cost of \$4.50 per DC ampere per month.

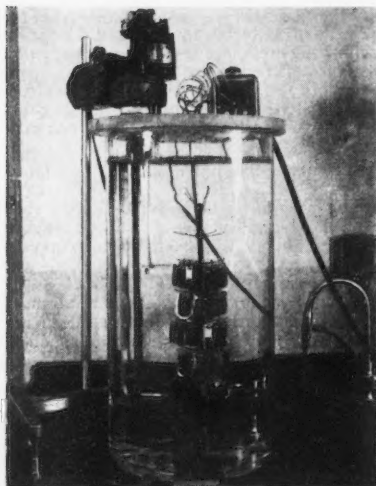


Figure 1—Test assembly.



Figure 2—Sensitized Type 304 stainless steel specimen after a 48-hour exposure to water containing 10 ppm Cl, pH 6-8, at 185 F. Specimen was in Run K. Note discolorations. 4X magnification with one-half reduction.



Scharfstein



Brindley

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# Chloride Stress Corrosion Cracking of Austenitic Stainless Steel— Effect of Temperature and pH\*

By L. R. SCHARFSTEIN and W. F. BRINDLEY

## Introduction

FIFTEEN YEARS ago, reported incidents of stress corrosion cracking of austenitic stainless steels were rare. The increased use of stainless steels during the past few years has resulted in a great rise in such reports. The major offenders have been aqueous chlorides. The tendency of stressed austenitic stainless steel to crack when exposed to chloride-bearing water at elevated temperatures (500 F) is well known.<sup>1,2,3,4,5</sup> However, only a few cases of chloride stress corrosion cracking in dilute solutions at temperatures below the boiling point of water have been reported.<sup>2,6,7,8</sup> This fact alone indicates that a reduction in temperature must lead to a reduction in the susceptibility of austenitic stainless steel to this type of attack. A test program was undertaken to determine the susceptibility of austenitic stainless steels to stress corrosion cracking in dilute sodium chloride solutions at the temperatures of 165 F and 200 F. The design of the test was prepared with the aid of the Bettis Statistical Staff.

Stressed U-bends were selected as corrosion specimens since experience has shown that this type of specimen serves as a convenient means of obtaining qualitative information.<sup>2,3,8</sup> In addition, the large amount of data collected with this specimen at 500 F<sup>2,3</sup> would be available for comparison with the results observed at the lower temperatures.

## Experimental

The tests were performed in a large cylindrical pyrex container measuring 24 by 12 by ½ inches. The container was fitted with a ¾-inch plexiglass cover containing a sampling tube, an electrically driven stirrer, an immersion heater, and a thermometer (Figure 1). The U-bend specimens were hung from a precorroded stainless steel stand.

AISI Types 304 and 347 stainless steel specimens were investigated. Since the objective of the test was to determine the maximum concentration of chloride incapable of initiating stress corrosion cracks in the U-bends at the test temperatures, a safe population sample size was required. For instance, the observation of one crack in only ten specimens could actually mean 460 cracked specimens if 1000 were tested with 95 percent confidence. On the other hand, if no cracks were observed in ten specimens,

## Abstract

Overstressed U-bends of Types 304 and 347 stainless steels were exposed to water containing chloride ions to determine the susceptibility of these steels to stress corrosion cracking between the temperatures of 165 F and 200 F. The pH was controlled at 6.5 to 7.5 and 10.6 to 11.2 for the tests. At the high pH, cracks appeared at the edges with little evidence of pitting. At the neutral pH, cracks were found at the edges and associated with pits. Sensitized Type 304 had longer and deeper cracks than annealed Types 304 and 347 in the same exposure time. Conclusion is made that chloride stress corrosion cracking of these steels in the temperature range of 165 F to 200 F is less severe than that experienced at 500 F and that specific conditions are required for corrosion cracking to occur at all.

3.2.2

as many as 330 cracked specimens might be the result if 1000 were tested.<sup>9</sup> A sample size of 10 specimens of each material per test run was used. If any cracks were found, the next lower concentration of chloride was to be tested until a level of chloride was found without causing cracks. To be certain of the latter finding, the run would be repeated several times and the results accepted as true if no cracks were found. As the test program progressed, it became apparent that cracking was initiated in less than 48 hours. The test period was therefore standardized at the 48-hour period.

The U-bend specimens were fabricated from strips of stainless steel measuring 3¼ by ¾ by ⅛ inches. Two ¼-inch diameter holes were drilled, each at a distance of ⅝ inch from the end of the specimen as measured along the centerline. These strips were machined from a hot rolled, annealed and pickled plate to yield a 16 microinches RMS or better surface finish. A piece of each heat of Type 304 stainless steel was sensitized by heating at 1200 F for two hours followed by air cooling. The sensitizing heat treatment was performed prior to machining. The strips were bent around a ⅝-inch radius mandrel until the legs were parallel. A thin strip of aluminum oxide paper was placed between the stainless steel strip and the brass mandrel to prevent contamination by the brass. Stress was applied by tightening the nut of a Type 304 stainless steel bolt and nut assembly placed through the holes of each specimen to a leg deflection of approximately 0.02 inch.

Each specimen was examined for surface defects by a fluorescent penetrant method (Magnaflux Super Pentrex, ZL-22) and by means of a binocular microscope at 30X magnification. The

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penetrant inspection served as a mass production method of eliminating grossly defective specimens. Final specimen acceptance was based on the microscopic examination. Specimens with evident nicks, gouges, scratches, rounded corners, etc., were rejected.

The specimens were suspended from the stand by means of a small stainless steel hook placed on the center of each bolt. Austenitic stainless steel bolts, nuts, hooks and stands were used to minimize the effects of galvanic currents. Individual specimens were separated by placing a small piece of austenitic stainless steel mesh between the specimens.

A normal test run was performed with 30 U-bend specimens and 34 liters of solution. The plexiglass cover was a tight fit in order to minimize evaporation during a run. The initial water level was marked and maintained by additions of distilled water, if necessary. No attempt was made to control dissolved gases. Therefore, the equilibrium oxygen concentration for each temperature was a test variable. The following oxygen concentrations were the average of more than three runs at each temperature: 165 F, 2.75 ppm  $O_2$ ; 185 F, 2.07 ppm  $O_2$ ; 200 F, 0.88 ppm  $O_2$ . Chloride was added as sodium chloride. All chemicals employed were certified reagent grade. Solutions were stirred at approximately 300 rpm and heat was applied by a 2500-watt Cromalox unit controlled to  $\pm 5$  F by a thermoregulator.

After the test unit was assembled, the heater was turned on and the solution temperature raised to the specified temperature. A complete chemical analysis for chloride, oxygen, pH, etc., was made after 24 hours and at the end of the run. At the completion of a run, the specimens were removed from the vessel and immersed in a flask containing deionized water. After rinsing, the specimens were air dried with clean acetone. Each specimen was examined by the penetrant and microscope techniques. Following the examination, each specimen was further stressed by deflecting the legs an additional 0.100 inch. This operation opened the existing cracks and helped make them more visible. Applied to untested specimens, the same technique failed to produce cracks, thereby proving the adequacy of the method. Crack indications were then sectioned and metallographically examined for verification.

Tables 1 and 2 list the heat numbers, certified analyses, and mechanical properties of each heat tested.

### Results

Table 3 summarizes the test conditions and results. Specimens exposed to neutral pH water (Runs A through M) exhibited numerous discolorations appearing as rust-colored spots. These spots were distributed randomly over the specimens (Figure 2) but were more numerous on the sides in tension. Figures 3 and 4 show typical stress corrosion cracks. All cracks were associated with a rust spot. Pitting was also found at discolorations in the runs performed at pH 6-8 (no chemical additives).

Early results indicated that the austenitic stainless steels were susceptible to chloride stress corrosion cracking at the relatively low temperatures of the test (Table 3). As the chloride level was reduced, the size of cracks decreased and examination became more difficult. The restressing of the specimens was the best aid in post-test examination. Little difference was found between the effect

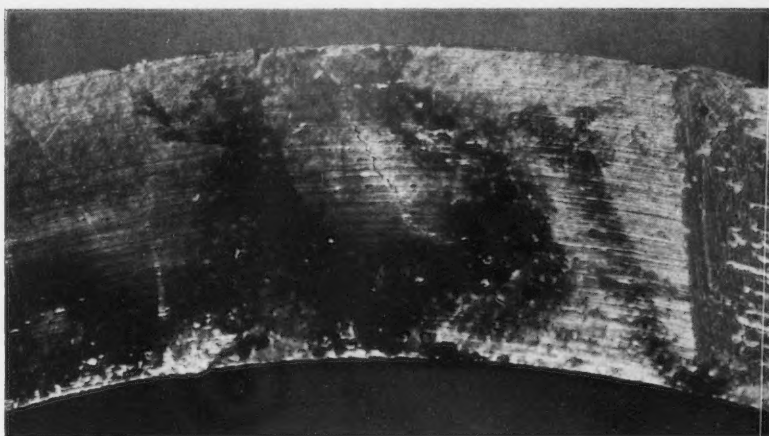


Figure 3—Crack seen in discolored area on sensitized Type 304 stainless steel specimen after a 48-hour exposure to water containing 220 ppm Cl, pH 6-8, at 200 F. Specimen was in Run D. 20X.

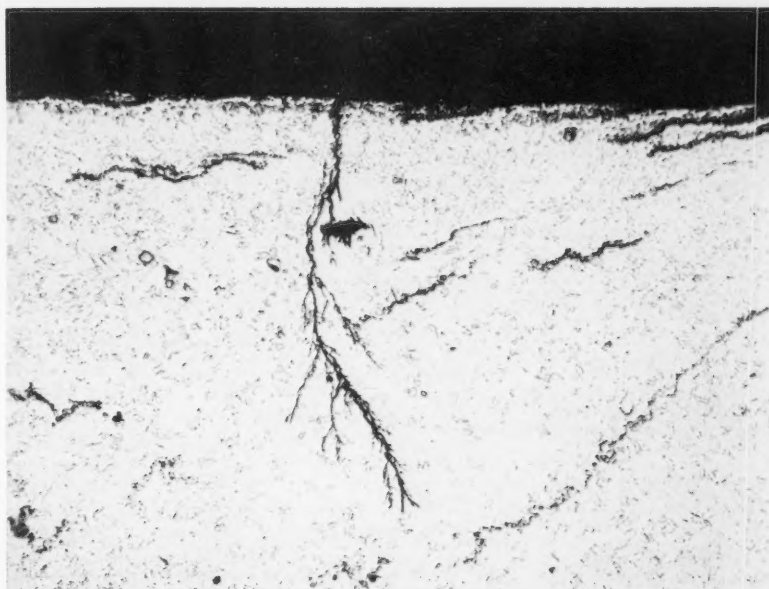


Figure 4—Photomicrograph of crack seen in Type 347 stainless steel specimen after a 48-hour exposure to water containing 25 ppm Cl, pH 6-8, at 200 F. Tested in Run M. 100X; etchant. Chromic acid, electrolytic.

of 165 F vs 200 F. At lower chloride concentrations, the solution at 165 F was slightly less aggressive than at 200 F. The addition of excess oxygen (Run L) increased the attack. This can be seen by comparing Run L with Run G.

Sensitized Type 304 stainless steel reacted somewhat differently than the "as received" steels. Cracks in the sensitized stainless steel (Figures 5 and 6) were usually predominantly intergranular. The unsensitized stainless steels cracked transgranularly. In general, the sensitized material showed deeper and longer cracks.

All specimens tested at the neutral pH without chemical additives had numerous pits. Since pitting of austenitic stainless steels in aqueous chloride reaches a maximum at 185 F,<sup>10</sup> Runs J and K were carried out at this temperature to test the effect of increased pitting. A marked increase in both pitting and cracking was observed. It was therefore

decided to perform the majority of the remaining tests at 185 F.

Previous tests performed with U-bends of austenitic stainless steel at 500 F have shown a tendency on the part of phosphate ion in alkaline media to inhibit chloride stress corrosion cracking. Likewise, pitting was rarely seen under these conditions. It was with this background that Runs O through S-2 were performed with  $Na_2HPO_4 \cdot 7H_2O$  and NaOH. Under these conditions, stress corrosion cracking was prevented at 185 F even with 550 ppm chloride. The 48-hour test period was considered sufficient since Runs G, H and I at 312, 523 and 480 hours respectively did not increase the number of specimens cracked. In these runs the specimens were initially examined after 48 hours and all uncracked specimens were returned to the solution. None of these specimens cracked during the remainder of the test interval. This result indicates that even at tem-

peratures of 165 F to 200 F stress corrosion cracking is a rapid phenomenon, occurring in less than 48 hours. In autoclave tests at 500 F, similar results were obtained with specimens cracking during the first hour at test temperature, and no increase in cracking was effected by extending the test period.

An unusual result of the runs made with alkaline phosphate was the effect of insufficient phosphate on the sensitized Type 304 U-bends. If the chloride concentration was high (550 ppm) and the phosphate concentration low (120

ppm), several severe pits were formed on the sensitized U-bends. Figures 7 and 8 show these pits. Under these conditions, the pits developed only in the sensitized steel. None of the specimens had cracks. An increase in the phosphate concentration, approximately equimolar with the chloride ion (Run P), resulted in all specimens free of pits and/or cracks. This result is interpreted to indicate the peculiar influence of the phosphate ion and not the pH rise alone since in both runs (high and low phosphate) the pH was 11.0 adjusted with

sodium hydroxide. Safe inhibition by the phosphate ion was also found in Runs Q and R where the phosphate ion concentration was adjusted to be equimolar with the chloride ion. Runs S-1 and S-2 were made to determine whether the pitting of the sensitized stainless as a result of insufficient phosphate could be arrested with an addition of phosphate. This did not prove to be true. Where severe pitting occurred in Run S-1, pit depths were measured and the specimens returned to Run S-2. The pit depths of 15 out of 16 cases increased as shown in Table 4.

Since the addition of disodium phosphate plus sodium hydroxide is equivalent to the addition of trisodium phosphate alone (assuming a negligible excess of sodium hydroxide), two tests were run (T and U) with the addition of trisodium phosphate directly. Results were good and the method is simple. A possible disadvantage is the lack of pH control. Large quantities of phosphate, added as trisodium phosphate, will raise the pH over 11.0 and possibly over 12.0. This may be detrimental to some mixed metal systems.

To test the effect of alkaline pH, a run (V) was made with the pH raised to 8.8 by sodium hydroxide alone. Under similar conditions without sodium hydroxide (Runs J and K), cracking in 70 to 100 percent of the specimens was noted. The run with sodium hydroxide resulted in no specimen cracking (no cracks observed in 30 specimens). The results of this run are interpreted to illustrate the inhibiting influence of the alkaline addition. Table 3 shows the pH drift with time presumably as a result of carbon dioxide dissolution. After approximately 100 hours, the pH had dropped to 7.0. The specimens were removed every 48 hours for examination and then were returned to the solution. Specimens did not crack even though they were returned to a solution with pH close to 7.0. This result is similar to those found in Runs G, H and I

TABLE 1—Chemical Analyses of Materials Tested.

Heat Number	Type Steel	COMPOSITION, PERCENT									
		C	Mn	P	S	Si	Ni	Cr	Cu	Cb	Ta
334220	304	.053	1.53	.021	.019	.43	9.15	18.40	...	...	...
3X8946	304	.033	1.07	.011	.011	.61	9.36	18.34	...	...	...
13636	304	.07	1.41	.030	.005	.74	9.80	18.60	...	...	...
3X8965	304	.038	1.29	.021	.015	.53	12.07	18.64	...	...	...
16104	304	.088	1.42	.012	.014	.69	9.63	18.54	...	...	...
13205	304	.055	.96	.032	.030	.34	9.20	18.50	...	...	...
46324-1A	347	.054	1.63	.022	.023	.55	11.23	18.76	.27	.78	.05
64319-1D	347	.059	1.33	.019	.023	.48	11.22	18.35	.24	.60	.12
33571	347	.055	1.72	.028	.015	.44	11.03	18.60	...	...	...
16837	347	.037	.88	.020	.015	.37	10.40	17.68	...	.50	.21

TABLE 2—Mechanical Properties of Materials Tested.

	Heat No.	334220	13205	46324-1A	64319-1D	33571	16837
		304	304	347	347	347	347
Tensile Strength (psi)		82,730	80,000	86,600	88,000	87,800	89,000
Yield Strength (psi)		36,310	30,000	49,000	40,000	43,000	42,000
Percent Elongation in 2 inches		60.5	63.0	51.0	55.0	50.0	59.0
Percent Reduction in Area		69.2	67.0	49.5	59.0	...	...
Hardness (Brinell)		137	...	...	...	...	163/170
Rockwell B		...	75/78	...	...	...	...
Grain Size		4-6	...	...	...	...	...

Heat numbers 3X8946, 13636, 3X8965, and 16104 were experimental heats produced by the U. S. Steel Company. No mechanical properties were reported.

TABLE 3—Test Results.

Run No.	ppm Cl (NaCl)	Temp. degrees F	pH	304 AS RECEIVED		347 AS RECEIVED		304 SENSITIZED		Time, hr.	Remarks
				Heat No.	% Cracked	Heat No.	% Cracked	Heat No.	% Cracked		
A	550	200	6-8	334220	10	46324-1A	20	334220	70	48	
B	550	165	6-8	334220	20	46324-1A	90	334220	40	48	
C	200	165	6-8	334220	0	46324-1A	80	334220	50	48	
D	200	200	6-8	334220	30	46324-1A	80	334220	50	48	
E	100	165	6-8	334220	0 <sup>1</sup>	46324-1A	0 <sup>1</sup>	334220	0 <sup>1</sup>	48	
F	50	200	6-8	334220	50	46324-1A	100	334220	10	48	
G	10	165	6-8	3X8946	10	64319-1D	100	334220	30	312	
H	5	200	6-8	3X8946	14	64319-1D	100	3X8965	38	523	7 Type 304 as received, 8 Type 304, 10 Type 347 specimens tested.
I	25	165	6-8	3X8946	20	64319-1D	60	3X8965	30	480	
J	25	185	6-8	3X8946	70	64319-1D	100	3X8965	100	48	
K	10	185	6-8	3X8964	90	64319-1D	100	3X8965	100	48	
L	10	165	6-8	3X8946	70	64319-1D	100	3X8965	60	48	Oxygen bubbled through to yield 16 ppm.
M	25	200	6-8	3X8946	90	64319-1D	90	16104	100	48	
N	0	185	6-8	13636	0	33571	0	13636	0	48	
O	550	185	10.8-11.0	13636	0	33571	0	3X8946	0 <sup>2</sup>	48	120 ppm PO <sub>4</sub> as Na <sub>2</sub> HPO <sub>4</sub> + NaOH
P	550	185	10.8-11.0	13636	0	33571	0	3X8946	0	48	1475 ppm PO <sub>4</sub> as Na <sub>2</sub> HPO <sub>4</sub> + NaOH
Q	200	185	10.8-11.0	13636	0	33571	0	3X8946	0	48	535 ppm PO <sub>4</sub> as Na <sub>2</sub> HPO <sub>4</sub> + NaOH
R	50	185	10.8-11.0	13636	0	33571	0	3X8946	0	48	150 ppm PO <sub>4</sub> as Na <sub>2</sub> HPO <sub>4</sub> + NaOH
S-1	550	185	10.8-11.0	13636	0	33571	0	3X8946	0 <sup>2</sup>	72	100 ppm PO <sub>4</sub> as Na <sub>2</sub> HPO <sub>4</sub> + NaOH
S-2	550	185	10.8-11.0	13636	0	33571	0	3X8946	0 <sup>2</sup>	48	1475 ppm PO <sub>4</sub> as Na <sub>2</sub> HPO <sub>4</sub> + NaOH
T	50	185	10.7-11.4	13636	0	33571	0	3X8946	0	48	200 ppm PO <sub>4</sub> as Na <sub>3</sub> PO <sub>4</sub>
U	550	185	11.7-12.0	13636	0	33571	0	3X8946	0	48	1475 ppm PO <sub>4</sub> as Na <sub>3</sub> PO <sub>4</sub>
V	10	185	8.8-7.0	13205	0 <sup>3</sup>	16837	0 <sup>3</sup>	3X8965	0 <sup>3</sup>	192	pH originally adjusted with NaOH
W	10	185	8.7-7.7	13205	0 <sup>3</sup>	16837	0 <sup>3</sup>	3X8965	0 <sup>3</sup>	204	1000 ppm K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> added
X	100	185	8.4-7.7	13205	0 <sup>3</sup>	16837	0 <sup>3</sup>	3X8965	0 <sup>3</sup>	192	1500 ppm K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> added
Y	550	185	8.1-7.8	13205	0 <sup>3</sup>	16837	0 <sup>3</sup>	3X8965	0 <sup>3</sup>	192	1000 ppm K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> added

<sup>1</sup> Not stressed additional 0.1 inch, rerun in G.

<sup>2</sup> All sensitized Type 304 SS specimens had deep pits.

<sup>3</sup> Specimens had tiny pits and rust spots.

<sup>4</sup> Rust spots, no pits.

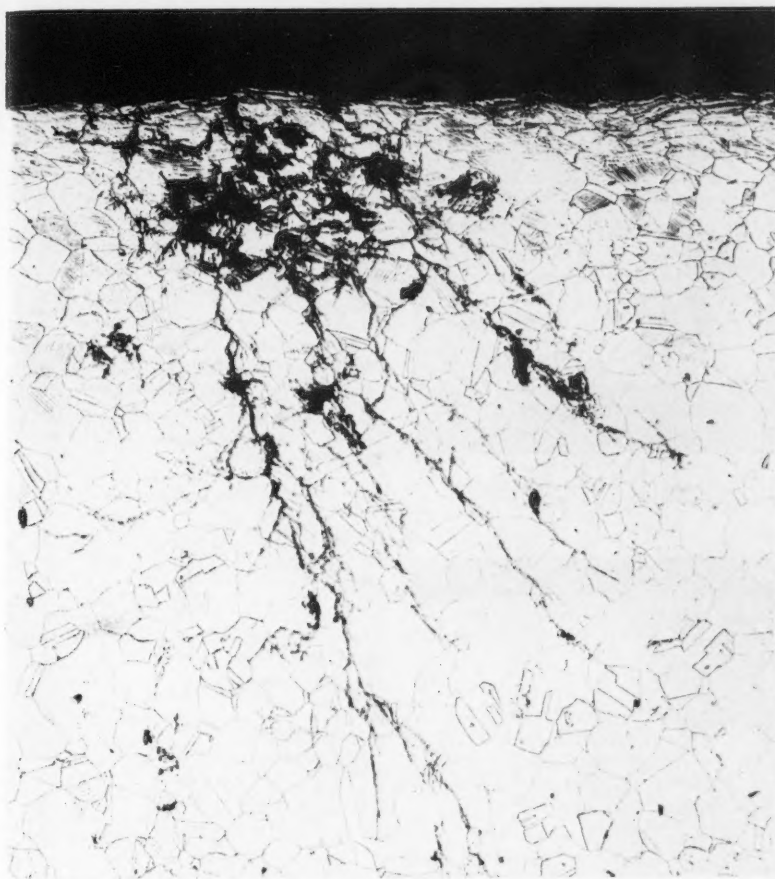


Figure 5—Transverse section through crack in sensitized Type 304 stainless steel specimen after a 48-hour exposure to water containing 200 ppm Cl, pH 6-8, at 165 F. Specimen was in Run C, 100X; etchant: chromic acid, electrolytic.

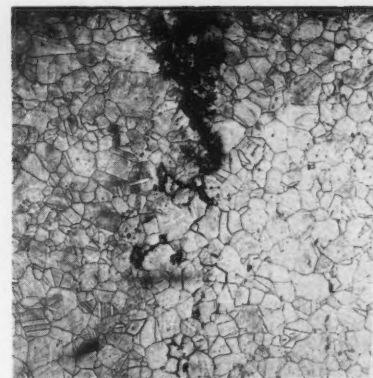


Figure 6—Transverse section through crack in sensitized Type 304 stainless steel specimen after a 48-hour exposure to water containing 200 ppm Cl, pH 6-8 at 200 F. Tested in Run D. 100X magnification with one-half reduction.

chloride stress corrosion cracking of austenitic stainless steel at low temperatures with the addition of chromate is interpreted to be more an effect of pH than anion inhibitor. The important point, however, is the fact that chromates are not accelerators under these conditions. This means that chromates can be added to mixed metal systems operating at low temperatures to prevent general corrosion without accelerating chloride stress corrosion attack of stainless steel.

#### Discussion

The results of the test program are interpreted to indicate that austenitic stainless steels are susceptible to chloride stress corrosion cracking in aqueous solutions if the temperature is even as low as 165 F to 200 F. The conditions which give rise to cracking, however, are specific. The pH must be close to 7.0 when the bare metal is exposed to the chloride-bearing water. Since the stainless steels do not crack severely under the low temperature conditions, cracking is much more easily prevented than at high temperatures (500 F). This observation is based on the fact that not all specimens crack, even at pH 7.0. Specimens exposed to the water, without cracking, do not crack on repeated exposure. This indicates the formation of a protective corrosion film. This simple pretreatment seems sufficient to prevent cracking. The same result was observed by adding sodium hydroxide to yield an initial pH of 8.8. All these specimens did not crack even after the pH dropped to 7.0. The addition of potassium chromate also prevented cracking but a survey of literature data<sup>4</sup> gave the authors the opinion that the result was simply due to the hydrolysis yielding pH of 8.8 and not to an inhibiting influence of the chromate anion. Phosphate, however, acts as an inhibiting anion for sensitized stainless steel. An insufficient ratio of phosphate to chloride ions resulted in a few relatively deep pits in the sensitized stainless steel. The addition of sodium hydroxide or hydrolyzing salts alone prevents cracking in all materials tested, but small pits were still observed on the stressed specimens.

#### Conclusions

Cold worked and stressed austenitic stainless steels are susceptible to chloride stress corrosion cracking at tem-

TABLE 4—Pit Depth Measurements of Sensitized Type 304 Stainless Steel\*

Specimen No.	PIT DEPTHS (Mils)					
	After First 72 Hrs.			After Additional 48 Hrs.		
	Pit No. 1	Pit No. 2	Pit No. 3	Pit No. 1	Pit No. 2	Pit No. 3
G-1678.....	5.75			12.30		
G-1685.....	6.41			6.75		
G-1683.....	18.05	Unable to Measure		33.10	0.98	
G-1679.....	12.56			15.35		
G-1684.....	12.13	4.16	1.77	20.54	4.68	3.19
G-1681.....	7.05	8.88	17.52	10.32	13.45	23.37
G-1686.....	5.82	13.17	Unable to Measure	6.33	19.21	2.80
G-1682.....	13.18			19.56		
G-1687.....	6.56			4.74		

\* Specimens were exposed to water containing 550 ppm chloride and 100 ppm phosphate for 72 hours, then to water containing 550 ppm chloride and 1475 ppm phosphate for 48 hours.

where specimens not cracking in the first 48 hours did not crack later regardless of time. This effect is attributed to the formation of a protective film on the specimens which is not damaged by the relatively non-corrosive environment.

One more additive was investigated in this program. Edleau<sup>1</sup> had shown that at boiler temperatures chloride stress corrosion cracking of austenitic stainless steel occurred even without oxygen if an oxidizing agent such as potassium chromate was present. To investigate the effect of chromate under low temperature conditions, three runs

(W, X, Y) were performed with potassium chromate additions. No cracks were found. There were numerous rust spots, but these did not contain pits or cracks. Since potassium chromate hydrolyzes in water to yield an alkaline reaction, tests with potassium chromate yielded an initial pH of approximately 8.8. The pH drifted downward with time, finally leveling at pH 7.7. Here again, specimens removed from solution and returned did not crack regardless of the downward drift in pH. Although chromates are considered to be general corrosion inhibitors, the prevention of



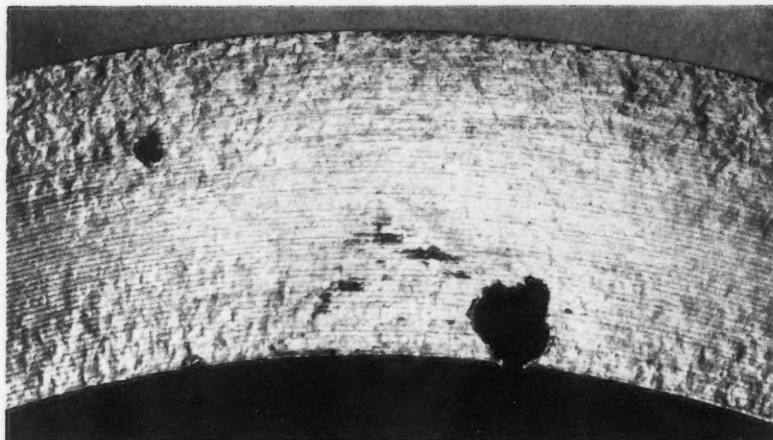


Figure 7—Sensitized Type 304 stainless steel specimen after a 48-hour exposure to water containing 550 ppm Cl, 120 ppm PO<sub>4</sub>, pH 10.8-11.0 at 185 F. Run was number O. Note deep pits. 20X.

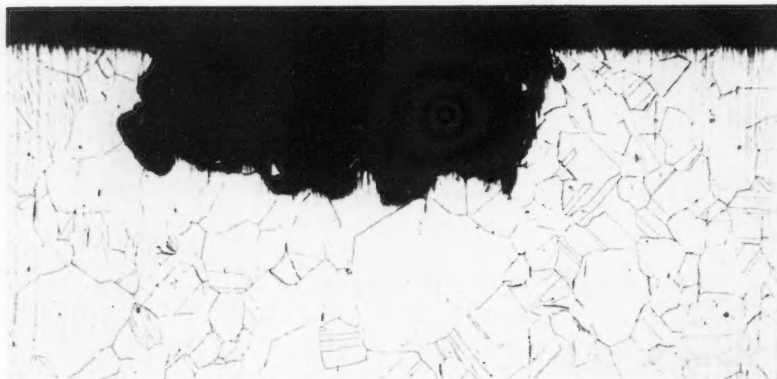


Figure 8—Photomicrograph showing pit seen in Type 304 stainless steel specimen after a 48-hour exposure to water containing 550 ppm Cl, 120 ppm PO<sub>4</sub>, pH 10.8-11.0, at 185 F. Tested in Run O. 100X.

peratures as low as 165 F to 200 F with as little as 5 ppm chloride in the water. There is little difference between the resistance of Types 304 and 347 stainless steel except where Type 304 is sensitized. The sensitized austenitic stainless steel cracks predominantly intergranularly and exhibits longer and deeper cracks than the unsensitized material. Unsensitized material cracks transgranularly.

The actual concentration of dissolved oxygen between 165 F and 200 F has a

slight influence on the results. This was proven by adding sufficient oxygen to raise the concentration over seven times. An increase in percentage of specimens cracked was found, but not all specimens cracked.

An increase in pH is beneficial probably through its reduction in pitting of the austenitic stainless steels in aqueous chloride. The addition of alkaline phosphate gave evidence of the inhibiting property of the phosphate ion. A concentration of alkaline phosphate, equi-

molar with the chloride ion concentration, gave complete protection both from cracking and pitting for all the austenitic stainless steels tested. The phosphate ion, however, was an unsafe inhibitor for sensitized austenitic stainless steel since an insufficient concentration led to localized pitting attack.

The addition of sodium hydroxide or potassium chromate was also beneficial. These results are concluded to illustrate the inhibiting influence of increased hydroxyl ion.

Pre-exposure of specimens without cracking, was sufficient pretreatment to prevent cracking when the pH was reduced.

The authors conclude that the chloride stress corrosion cracking of austenitic stainless steel in the temperature range of 165 F to 200 F is much less severe than experienced at 500 F and requires specific conditions for it to occur at all.

#### Acknowledgment

The authors wish to thank the United States Navy for their sponsorship of the test program. The authors also wish to express their appreciation to C. A. Walter for his assistance in examining specimens and to G. P. Hensler for his aid in carrying out the program and assembling this paper.

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Any discussions of this article not published above will appear in the June, 1959 issue



## DISCUSSIONS

**Cathodic Protection of Lead Cable Sheath in the Presence of Alkali From Deicing Salts by Walter H. Bruckner and W. W. Lichtenberger. Corrosion, Vol. 14, No. 4, 165t-170t, (1958) April.**

**Comment by V. E. Lowden, Illinois Bell Telephone Co., Chicago, Illinois:**

The authors are to be congratulated on the work reported here. The attempt to relate abstract theory to simulated field conditions is certainly laudable.

As the authors suggest, their conclusion that lead cables might be salted to increase their service lives comes as a shock to many of us in the field. We have observed a definite correlation between the presence of deicing salt and corrosion failures where the cables have been cathodic.

One of the most gratifying results reported in the paper was the production of actual corrosion on the lead cathodes. I believe that this is the first time cathodic lead corrosion has been obtained under controlled laboratory conditions, with perhaps one exception.\* It is to be hoped that the authors will explore this matter further so that eventually we will understand exactly what happens to cathodic lead sheath when it is exposed to deicing salt solutions.

**Reply by W. H. Bruckner and W. W. Lichtenberger:**

We are pleased to have Mr. Lowden's comments and to have him express his surprise that deicing salts could be anything but a disadvantage to a cathodic protection system for lead cable sheath.

The burden of the argument which we presented in our paper was that under precise control of the cathodic protection system with respect to constancy of current and full submersion, the presence of a uniform concentration of deicing salts would provide a more uniform distribution of cathodic current to the cable sheath. The unpleasant problems of maintaining a cathodic protection system on lead cable sheath in underground ducts arise because of the non-uniform distribution of deicing salts, the attendant fluctuation of current to the system and the seasonal variation in water level. Our paper has shown that it is possible to have the cable sheath suffer corrosion loss even while under cathodic protection when the relationship of the pH and potential was such as to be outside of a protective area as defined by the Pourbaix diagram. Since a duct system has maximum current input to the lead cable sheath at the joints or other openings, potential gradients will exist as simulated in the gradient test cells described in our paper. The data in our paper show that corrosion loss in such a gradient system occurs first at the part of the lead cathode most remote from

the point where current enters the system. However, these data were obtained for a constant current system whereas in practice the current rarely is constant.

New data established since the paper under discussion was written show that corrosion of lead cable sheath also can occur at the high potential end of the gradient cathode when the supply of current to the system is periodically varied. The reason for this occurrence is that the overprotected region of the cathode fails to develop a protective film; thus upon cessation of cathodic protection current the region has a maximum initial corrosion rate in an environment with maximum pH. The new data will be incorporated in a paper to be presented to the NACE in 1959. It should be noted that the experience of the field (namely, corrosion of a lead cable system at regions of maximum salting), has now been observed in the laboratory when the discontinuous current supply in service was simulated.

**Corrosion Rates of Mild Steel in  $\text{NH}_4\text{NO}_3\text{-NH}_3\text{-H}_2\text{O}$  Solutions by Norman Hackerman, Ray M. Hurd and Earl S. Snively. Corrosion, Vol. 14, No. 4, 203t-205t, (1958) April.**

**Comments by G. H. Cartledge, Oak Ridge National Laboratory, Oak Ridge, Tennessee:**

In Frumkins' laboratory it has been found from polarization measurements in the absence of oxygen that, at the same potential, the anodic (corrosion) reaction is  $10^6$  times faster in an alkaline solution than in an acidic solution. In a careful repetition of these measurements in Bonhoeffer's laboratory, it was found that the factor is  $10^8$ , instead of  $10^6$ . It is obvious that the  $\text{OH}^-$  by its adsorption, greatly lowers the activation energy of the anodic process, probably by formation of an intermediate complex. It may be suggested that in the ammonia system there may be a similar acceleration by amide ions ( $\text{NH}_2^-$ ), the adsorption of which may be prevented by the sulfur-containing inhibitor.

**Reply by Norman Hackerman:**

The authors wish to thank Dr. Cartledge for this interesting suggestion. While we did not make controlled measurements of the corrosion rate at definite potentials, it is clear that the rate is many orders of magnitude faster here than in the comparable acid system ( $\text{NH}_4\text{NO}_3$  in  $\text{H}_2\text{O}$ ). We also agree that the activation energy of the anodic process is greatly lowered, but we are not yet in a position to describe the mechanism. The amide ion adsorption hypothesis is a definite possibility. Experiments carried out after the subject paper was presented have shown that nitrite ion is formed during the corrosion process, but as yet we have not been able to identify the presence of  $\text{NH}_2^-$ .

**Question by Thomas L. Hoffman, Idaho Falls, Idaho:**

$4\text{NH}_4\text{NO}_3 \cdot 1\text{NH}_3 + 1\text{H}_2\text{O}$  (pH 9.5) is a specific solution. What quantity 40 percent caustic would be required to increase the pH to 14? Under these conditions of higher pH, would inhibition occur on any type mild steel such as SAE 1020?

**Reply by Norman Hackerman:**

The addition of 40 percent caustic to this solution would result in the liberation of  $\text{NH}_3$  to the extent that at pH 14 the solution would be essentially  $\text{NaNO}_3$  and  $\text{NaOH}$  water. The amount required would be that necessary to convert the  $\text{NH}_4\text{NO}_3$  to  $\text{NaNO}_3$ , drive off most of the  $\text{NH}_3$ , and then raise the pH of a  $\text{NaNO}_3$  solution to 14. Since the solution is now entirely different, we do not know whether or not mild steels would be inhibited by the thiocyanates, but we do not recall offhand any instances of thiocyanate inhibition in solutions of this type.

**Methods for Increasing the Corrosion Resistance of Metal Alloys by N. D. Tomashov. Corrosion, Vol. 14, 229t-236t, (1958) May.**

**Comments by W. F. Higgins, Magnesium Electron Limited, Manchester, England:**

I should like to compliment you on the publication of an outstanding article, and the author of a very well considered, and clearly thought out exposition of the subject. If there is little that is entirely new, the simple direct approach is very refreshing, and the arguments in general are unassailable. However my purpose in writing is not merely to add my tribute to the many which I am sure you will receive but to comment on certain details of the author's conclusions.

In Table 3, section 2, second part, c, I question whether the case of manganese and zinc in magnesium is a good example of bringing about an increase in cathodic over-voltage. It is true that manganese, and probably zinc also, offset the lowering of hydrogen over-voltage brought about by iron—possibly by shrouding the iron particles and thus forming a less effective cathode, but this is not quite the same thing. Manganese however is very much more effective in this respect than zinc, yet the over-voltage of zinc and manganese are practically the same (0.39 volt in 3 percent sodium chloride solution according to Hannawalt).\* It thus appears that some other explanation is required for the beneficial effect of manganese. Two things are noticeable in the corrosion of the binary alloy of magnesium and manganese. One is that the manganese seems to be chiefly effective when it is present in solid solution (apart from its blanket-

\* W. S. Woodward, Bronx, New York. Private communication.

\* Corrosion Studies of Magnesium and Its Alloys, Technical Paper No. 1353, American Institute of Mining Engineers.

ing effect on iron particles mentioned above). Once particles of manganese or manganese/iron are present corrosion resistance is much less marked, presumably because the manganese particles are themselves fairly effective cathodes. The other point is that there seems to be a kind of threshold value of corrosion severity below which corrosion is almost absent but above which it can be very rapid. Unlike most other magnesium-rich alloys the magnesium-manganese binary alloy in strongly corrosive conditions becomes subject to heavy attack at isolated points in an area otherwise quite free from corrosion. It would seem that the manganese plays the part of an anodic inhibitor, that is it inhibits attack at anodic points, and, like all anodic inhibitors, concentrates attack at weak points when the corrosion pressure becomes too severe for complete protection.

An alternative explanation is that the manganese passes into solution with the corroding magnesium and is redeposited as a gelatinous hydroxide on the cathodic areas. This latter view finds some support from the yellowish brown color which often makes its appearance on corroded specimens of the binary alloy.

If the second theory is true, then the use of manganese in magnesium alloying ought to find a place in Table 3 in part 2, Reduction of Microcathode Areas, c, by auto-blanking of freshly exposed cathodes. On the other hand if the first suggestion is the true explanation it should appear in the third section of the table as a further example, (d), in the group "Introduction of Constituents Increasing Passivation of the Anode Phase."

The above properties of manganese appear to be shared to some extent by zirconium.

I would like also to comment on the author's conclusion that reduction of the alloy anode activity offers the greatest possibilities of producing electrochemically corrosion resistant alloys and the example of the high strength aluminum alloys containing magnesium which he quotes in support of this. The statement is true of general corrosion but the very fact that the intergranular constituent is made anodic to the general background renders the alloy sensitive to intergranular attack. The method therefore would seem to result in exchanging one undesirable feature for another which may be even worse.

#### Reply by N. D. Tomashov:

The decrease of corrosion rate of technical magnesium contaminated by iron, associated with supplementary alloying with manganese, seems to have several causes. Eventual fixation of part of the iron in melted magnesium, due to the formation of the scarcely soluble intermetallic compound of iron and manganese (i.e., the refining of magnesium) is one factor. Such a mechanism is not essentially associated with the manganese remaining in solid alloy. It is known, however, that the increasing manganese content in previously prepared technical magnesium also augments its corrosion resistance. This augmenting could result because:

The presence of manganese in the solid solution hinders the normal course of anodic dissolution of magnesium, or the manganese in this particular case plays, as Mr. Higgins asserts, the part

of anodic inhibitor. Moreover, the manganese contained in technical magnesium can decrease the rate of its corrosional destruction through slackening of the cathodic process (increasing of hydrogen overpotential).

The cathodic braking, as brought about by manganese, can be accomplished in two ways:

(1) Secondary segregation from the solution in cathodic regions of Fe (or intermetallic compound Fe-Mg) in the form of colloidal hydroxides, or

(2) Direct formation of intermetallic compounds and solid solutions (Fe-Mg) or (Fe-Mg-Mn) having a considerably lower hydrogen overpotential as compared with the same metallic phases but without manganese.

On the receipt of more exhaustive quantitative data on the question about the principal influence of manganese could be reconsidered. At that time, as Mr. Higgins observes quite correctly, the example in question would take a new place in Table 3.

I agree that the reduction of the anodic phase area occasionally could result in the development of more dangerous local corrosion, such as intercrystalline or pitting corrosion. But our example of increasing the corrosion resistance of the high strength aluminum alloys with 5-9 percent Mg (not 59 percent as misprinted in the article) pertains to these cases of heat treatment, which assure the formation of more fine and clear-cut boundaries and reduce thereupon the development of intercrystalline corrosion of the alloy.

#### Corrosion of Steel in Water by Varied Ratios of Dissolved Gases by J. Wade Watkins and G. W. Kincheloe. Corrosion, Vol. 14, 341t-344t, (1958) July.

Question by Joe Chittum, Whittier, California:

We have obtained comparable results with  $H_2S-CO_2$  systems and were interested in the data presented in this paper. We were unable to make comparison between our results and those reported in the paper for various reasons, one of which is that the authors do not give the surface area factor. We think that the ratio of actual to measured surface area should be reported. Has the surface area factor been obtained?

#### Reply by J. Wade Watkins:

Surface areas of the corrosion-test specimens used for the experiments reported in the subject paper were calculated from measurements of the individual steel specimens and were not measured directly. Therefore, the ratios of actual to measured surface areas were not obtained. Resultant variations in the actual surface areas of different specimens might have resulted in slight errors in the calculated and reported corrosion of those particular specimens. However, the trend of results should have remained unchanged. Also, each point plotted represented the arithmetic mean of the corrosion caused to several test specimens, and the calculated variance between individual specimens was such that no gross errors in measurements were indicated.

#### Question by H. Lee Craig, Jr., Reynolds Metals Co., Richmond, Virginia:

Did you determine the effect of temperature on the corrosion rate in any preliminary experiments? I would like to call your attention to the work of Van Rysselberghe and Delahay on the action of oxygen and carbon dioxide on the corrosion of steel in aqueous solutions. They show the formation of percarbonic acid which is an active corrodant of steel. Is it possible that the hydrogen sulfide interferes in this reaction, perhaps by forming a persulfo-carbonic acid?

#### Reply by J. Wade Watkins:

The effect on corrosion rates of temperature changes over a limited range (i.e., about 50 to 120 F), was determined before parameters were established for the described experiments. Increases in corrosion were reasonably linear with increases in temperature over the cited range. The operating temperature of 80 F was arbitrarily selected as representing a reasonable temperature for injection water under field conditions.

The reference to the work on Van Rysselberghe and Delahay has been noted with interest. In the Bureau of Mines experiments a definite lessening of the corrosion rate caused by dissolved free carbon dioxide occurred in the presence of dissolved hydrogen sulfide. Although several reactions were considered as possible causes of the exhibited inhibiting effect of hydrogen sulfide, the cause has not been established and logically might be the subject of additional research.

#### The Corrosion Behavior of Zirconium-Uranium Alloys in High-Temperature Water by Warren E. Berry and Robert S. Peoples. Corrosion, Vol. 14, 414t-418t (1958) Sept.

Question by Cornelius Groot, Richland, Washington:

The poor corrosion resistance of high uranium alloys in the uranium-zirconium system was explained as an inherent property of the  $\alpha$  uranium phase. One would expect any high uranium alloy to contain an  $\alpha$  uranium phase. Does this imply that all high uranium alloys must have poor corrosion resistance?

#### Reply by Warren E. Berry and Robert S. Peoples:

All high-uranium alloys do not necessarily contain an alpha-uranium phase and, hence, have poor corrosion resistance. Several alloys, notably those containing 12 percent molybdenum or 10 percent niobium, are sufficiently corrosion resistant for reactor applications when rapidly quenched from the high-temperature gamma phase (solid solution of the two elements). These alloys eventually fail by cracking from internal hydriding, as described by Burkhart and Lustman.\* Alloys containing lesser amounts of niobium possess a retained alpha structure upon rapid quenching from the gamma phase field. This structure also is corrosion resistant in high-

\* Corrosion Mechanism of Uranium-Base Alloys in High-Temperature Water. Transactions of Metallurgical Society of AIME, 212, 1, p. 26 (1958) Feb.



temperature water but slowly transforms to alpha uranium and is completely oxidized within two to three weeks' exposure at 600 F (315 C).

The zirconium alloys in this program were studied in the annealed condition. As explained in the paper, this condition would be least likely to result in cracking-type failure. However, the gamma phase can be retained by rapid quenching of alloys containing 20 through 80 percent uranium. In alloys of more than 60 percent uranium, the gamma phase transforms to the less resistant alpha uranium at the test water temperature and the alloys are rapidly oxidized. In alloys containing 20 to 50 percent uranium, the transformation to the equilibrium phases of epsilon plus alpha zirconium occurs more slowly. The hardness increase from the transformation frequently results in cracking of these alloys.

**Comments by R. D. Misch, Argonne National Laboratory, Lemont, Illinois:**

The authors have made a valuable contribution to existing information on Zr-U alloy corrosion, especially in relating corrosion to microstructure.

Besides the phase diagram of the metals some attention should also be given to phase relationships in the oxides.  $ZrO_2$  and  $UO_2$  have an extensive range of solid solutions which can be attributed to their favorable cation sizes. However, the protectiveness of a  $ZrO_2$  film is much greater than that of a  $UO_2$  film. The conductivity of the latter oxide suggests that it can accommodate many more lattice defects than  $ZrO_2$ . If a solution of the oxides is formed on an alloy surface then the defects normally present in  $UO_2$  will be diluted by the more stable  $ZrO_2$ .

The role of oxygen can be understood primarily as precipitating zirconium from its solution in the epsilon phase. The corrosion film on the depleted areas will be less protective and permit localized penetration.

Other elements which could decompose epsilon by removing zirconium would also be dangerous. Carbon and nitrogen probably act in this manner.

Information on the compositions of the corrosion films would be interesting, if available.

**Reply by Warren E. Berry and Robert S. Peoples:**

The comments by Dr. Misch are both interesting and enlightening. Our corrosion program did not include a study of the nature or growth of the corrosion films on the zirconium-uranium alloys. We did not obtain information on the compositions of the corrosion films.

**Comments by S. Kass and K. M. Goldman, Westinghouse Electric Corp., Bettis Plant, Pittsburgh, Pa.:**

The authors are to be complimented for their fine work on studying and interpreting the corrosion behavior of zirconium-uranium alloys in high temperature water. In general, the conclusions reached by Berry and Peoples are in agreement with work reported by others.<sup>1,2,3,4</sup>

The major portion of our discussion is concerned with the general effect of heat treatment on the corrosion resistance of various compositions (except single phase epsilon alloys) in the Zr-U

system. It is certainly understandable why the authors chose to study only the equilibrium structures. It is of interest to point out, however, that heat treatment does have a pronounced effect on many of the alloys in this system.

It has been reported that the 680 F water corrosion behavior of zirconium alloys containing 9 and 15 weight percent uranium is markedly affected by prior thermal treatment.<sup>1</sup> The data presented in Table 1 show the influence of heat treatment on these alloys.

It is noted that the specimens representing the 9 weight percent uranium alloy are most corrosion resistant (i.e., exhibit adherent growing oxide films) after vacuum annealing for 24 hours at 550 C and air cooling. Anomalous behavior is noted for the 15 weight percent alloy, where the more corrosion resistant form was obtained after vacuum annealing at 950 C and quenching.

Additional experiments with sponge zirconium base alloys containing 10 to 20 weight percent uranium showed that the "equilibrium condition" is the least corrosion resistant form of these alloys.<sup>2</sup>

The behavior of the 22 weight percent uranium sponge zirconium and high oxygen sponge base alloys, alternately showing adherent and nonadherent oxides, is different from that noted at Bettis where specimens from two 15 lb sponge zirconium base alloys containing 20 weight percent uranium corroded linearly in 600 F and 680 F water. Have the authors investigated the soundness or homogeneity of their 22 percent uranium ingot?

It is very interesting that the techniques for obtaining the authors' corrosion data were based upon the original area measurements. Perhaps the authors could explain the techniques for calculating their corrosion rates, taking into consideration the changes in area due to metal loss from corrosion.

There is yet another point we would like to mention. That is the variation of corrosion rate with uranium content. Results of work at Bettis, not yet published, indicate that the rate of corrosion for a given heat treatment increases linearly up to about 50 weight percent uranium after which it approaches an exponential behavior. Your data indicate that, for sponge base alloys, the rate appears to level-off with uranium content beyond 30 weight percent uranium. Would you care to comment on this point?

<sup>1</sup> Vacuum annealed 1 hour at 800 C, quickly cooled to 500 C, then furnace cooled to room temperature.

**TABLE 1—Weight Change of Alloys in mg/dm<sup>2</sup> After 56 Days in 680 F Water (Kass and Goldman Discussion)**

Weight Percent Uranium	Weight Percent Zirconium	950 C Quench	950 C Furnace Cooled	Air Cooled from 550 C
9.....	Bal.	-7461	-5095	+ 589
15.....	Bal.	-1600	-8955	-8020

**TABLE 2—Corrosion Rate of Alloys in mg/cm<sup>2</sup>/hr (Kass and Goldman Discussion)**

Percent Uranium	575 C FC*	575 C Q**	850 C Q	850 C FC	900 C Q	850 C Q + 10hr 575 C FC
10.....	-0.03	-0.03	+0.001	-0.04	+0.001	-0.03
15.....	-0.05	-0.05	+0.008	-0.06	+0.005	-0.07
20.....	-0.08	-0.08	+0.015	-0.09	+0.018	-0.08

\* Furnace Cooled.

\*\* Quench.

## References

1. S. Kass. The Corrosion Properties of Zirconium Base Fuel Alloys. Paper No. 57, Second Nuclear Engineering Science Conference.
2. S. Kass and K. M. Goldman. The Aqueous Corrosion of Zirconium Alloys Containing 10 to 60 w/o Uranium. In press (unpublished).
3. S. Kass and K. M. Goldman. The Corrosion Behavior of Uranium-Zirconium Alloys in High Temperature Water. WAPD-T-416.
4. R. K. McGeary, et al. WAPD-127, Pt. 1.

**Reply by Warren E. Berry and Robert S. Peoples:**

We have found in our work at Battelle that heat treatment affects the corrosion behavior of zirconium alloys containing less than 20 weight percent uranium. However, the effect has been the reverse of that presented in Table 1. It has been our experience that alloys in this composition range are more corrosion resistant when rapidly quenched from the high temperature phase field than when furnace cooled or transformed at temperatures of 500 to 600 C.<sup>1</sup> The quenched alloys transform martensitically and are more corrosion resistant than the furnace cooled or transformed alloys in which structures approach equilibrium.

It is unusual that the zirconium-9 weight percent uranium alloy, which was annealed 24 hours at 550 C, as described in Table 1 should gain weight. The structure of this alloy should approach equilibrium epsilon and alpha zirconium. It has been our experience that alloys with this structure do not form adherent films but lose weight in corrosion. The results presented by Kass and Goldman in Table 2 are comparable with those obtained in our work at Battelle. The alloys which were quenched from the high temperature phase field were more resistant and exhibited adherent films and gained weight. Those which were transformed by furnace cooling from above 850 C or annealing at 575 C were not so resistant and lost weight. The duplication of corrosion results presented in Table 2 for alloys with similar structures is excellent.

In reply to the question concerning the 22 weight percent uranium ingot, the ingot appeared to be sound and homogeneous. The build-up and spalling of the oxide on these alloys appeared to be related to the finely divided microstructure.

In reply to the question concerning the methods for calculating corrosion rates based on the changing area due to metal loss from corrosion, the technique was as follows: Samples were measured periodically during corrosion testing. An

average area was determined for each exposure period and a corrosion rate was calculated based on the average apparent area and weight loss for each period. Apparent areas changed as much as 10 to 20 percent on the higher uranium alloys after several thousand hours of exposure in 680 F water.

The linear increase in corrosion rate with increasing uranium content reported by Messrs. Kass and Goldman is not inconsistent with the results presented in this paper. The corrosion behavior of zirconium alloys containing up to 50 percent uranium apparently is determined by the amount and distribution of phases in the alloy. The latter are influenced by several factors: uranium content, oxygen content in the alloys, and heat treatment and prior fabrication history. In this paper, it has been possible to account for anomalies in corrosion results by examination of microstructures. Examination of the microstructures of the alloy series of Messrs. Kass and Goldman may indicate the reasons for the linear increase in corrosion rates with increasing uranium content.

#### Reference

1. H. A. Pray and W. E. Berry. Corrosion of Uranium Alloys in High Temperature Water. *BMT-874* (1953) Oct. 5.

#### Removal of Corrosion Products from High Temperature, High Purity Water Systems With an Axial Bed Filter by R. E. Larson and S. L. Williams. *Corrosion*, Vol. 14, 424t-428t, (1958) Sept.

Comments of W. L. Pearl, General Electric Co., Atomic Power Equip. Dept., San Jose, Calif.

We believe the development program reported by Messrs. Larson and Williams has been very worthwhile. We are looking forward to obtaining actual plant performance data to permit a more accurate sizing of the filter and resin beds to properly accomplish the objectives.

We have had occasion to evaluate what the dollar savings would be in the utilization of this magnetite filter principle for a central station nuclear power plant.

Let us take the case of a high temperature water nuclear power plant (boiling or pressurized) of about 200 MW electrical capacity. A continuous cleanup flow rate at about 100,000 lb/hr (200 gpm) would be the right order of magnitude to maintain a residual total solids content of about 0.5 ppm in the recirculating coolant with a mixed-bed demineralizer cleanup at expected system corrosion rates.

Utilizing Type 304 stainless steel construction, the major pieces of equipment required for an adequately sized cleanup system would be assessed costs as follows:

Demineralizers	\$ 60,000
Regenerative heat exchangers	40,000
Non-regenerative heat exchangers	40,000
Canned rotor recirculating pumps	25,000
Total	\$165,000

In the cooling and reheating of the 100,000 lb/hr of water, a net temperature differential of about 100 F between

No.	Material	Year Buried	Year Excavated	Duration of Test, Years	Maximum Penetration of Corrosion (in Millimeters)
584	(Ductile iron).....	1951	1954	3	1.2-1.2-1.1-1.1
583	(1st pipe man).....	1951	1957	6½	1.9-1.9-1.8-1.7
635	Unalloyed ductile iron 2 samples..	1954	1957	3½	1.2-1.1-1-0.9-0.9
640	Unalloyed ductile iron 2 samples..	1954	1957	3½	0.6-0.6-0.5-0.5-0.5
647	Low-alloyed ductile iron.....	1954	1957	3½	1-0.9-0.9-0.9-0.9
594	Semi-phosphorous gray iron of current manufacture.....	1954	1957	3½	1.3-1.3-1.2-1.2-1.2
		1951	1957	6½	2.2-2-1.9-1.8-1.8

the influent and effluent of the cleanup system would be expected. Considering only the net cost of the fuel as being wasted (at the rate of 3 mills per kw hr) and two refills of resin per year, the total annual operating cost would be over \$25,000 per year.

We will assume that this system could be replaced by a comparable sized magnetite bed filter with a small flow demineralizer unit to accomplish soluble fission product removal.

The demineralizer tanks could be used for the magnetite bed container at flow rates of 12-15 gpm/sq ft. The magnetite could be purchased commercially for about one-tenth the cost of the comparable resin. The entire equipment cost for such an installation would probably be under \$65,000. The small demineralizer system (about 25 gpm) would also be about \$25,000 installed.

The operating cost of the magnetite bed filter and small demineralization installation would be nominal compared to the large demineralization system since low heat losses are involved and the cost of replacement of magnetite is only about one-tenth that of the resin.

From the above rough calculation it appears that investment cost savings of the order of \$75,000 and operating costs of \$20,000 per year can be realized if such a magnetite bed filter can adequately provide the cleanup for a large nuclear central power plant.

#### The Corrosion Resistance of Ductile Iron by F. L. LaQue. *Corrosion*, Vol. 14, 485t-492t, (1958) Oct.

Comments by R. W. Manuel, Socony Mobil Oil Co., New York, N. Y.:

The "graphitic corrosion product" layer on cast iron, as pointed out in the paper, consists of more than graphite flakes. Microscopic examination sometimes reveals a structure in this layer similar to that of the parent metal, with graphite flakes, phosphide eutectic, and lamellar carbides of the pearlite all in place. Only the ferrite is corroded away, leaving sufficient residue of oxides and silicious compounds to hold the mass together. It would be interesting to know whether the graphitic corrosion product of ductile iron also has a pseudomorphic structure compared with its parent metal. Silicon may be important for furnishing corrosion products which aid in cementing the other residues together. Since ductile iron and gray cast iron have similar silicon content, they would be expected to resemble each other more than either would resemble malleable iron in corrosion behavior.

TABLE 2 (Paris Discussion)

Material	Duration of Test, Years	Maximum Depth of Pits, Millimeters
Centrifugally cast gray iron....	5½	0.8
Unalloyed ductile iron.....	5½	0.8
Low Ni alloyed ductile iron....	5½	1
Centrifugally cast gray iron....	3	0.9
Unalloyed centrifugally cast ductile iron.....	3	0.5
Low-alloyed centrifugally cast ductile iron.....	3	0.7

#### Reply by F. L. LaQue:

We have not, as yet, accumulated evidence to show that a graphitic corrosion product layer on ductile iron has a pseudomorphic structure such as is frequently observed with similar corrosion products on gray iron. We agree that the silicon in ductile iron will contribute to the consolidation of such corrosion products in ductile iron, and that, for this reason, ductile iron is more likely to behave corrosion-wise like gray iron than like malleable iron.

#### Comments by Michel Paris, Societe des Fonderies de Pont-a-Mousson, Nancy, France:

We wish to add some supplementary information on two points put forth in Mr. LaQue's paper. The first point involves underground corrosion.

#### Underground Corrosion

Since the conducting of tests in the two aggressive locations of Bay du Mont Saint-Michel and Touquet mentioned by Mr. LaQue, we have carried out more tests of longer duration. These tests were made on two meter-long pipe sections buried in an aggressive soil to such a depth that they were at the edge of the zone flooded by sea water. The tests lasted from 3 to 6½ years.

Table 1 compares the results with those from the very clayey soil of Touquet. The same comparisons are made with Mont Saint-Michel in Table 2.

The resistance to corrosion in these tests is almost the same for the different metals, with a slight advantage shown for unalloyed ductile iron over low alloy ductile and gray iron.

#### Corrosion of Petroleum Tankers

The second point in question concerns corrosion of petroleum tankers.

We do not think that the role of cathodic protection in the case of steel in contact with ductile iron has been very great. We do not believe, either, that it can explain the better behavior of Ni alloyed ductile iron.

We attached to the bottoms of two



**TABLE 1—Corrosion Rates of Alloys 3003 and 5052 At Two Locations\* (Godard Discussion)**

ALLOY	CORROSION RATES—mpy			
	Atlantic (Halifax, N.S.)		Pacific (Esquimalt, B.C.)	
	1 Year	2 Years	1 Year	2 Years
3003.....	0.35	0.07	0.13	0.08
5052.....	0.13	0.07	0.15	0.11

\* From Reference 2.

petroleum tankers making the run from the Persian Gulf to Eastern Europe a number of samples of piping of about ten different irons. In one of these tankers the samples were electrically isolated from their attachments and from the rest of the ship.

Without the use of cathodic protection we proved the excellent behavior of Ni alloyed ductile iron and its superiority over the unalloyed iron. We do not believe that the role of cathodic protection supplied by steel components in the case of heating pipes was important.

In the case of petroleum tankers transporting crude from the Persian Gulf to Eastern Europe, we believe the superiority of Ni alloyed ductile iron is indisputable. The heating pipelines fabricated with this iron and buried in 1952 showed no traces of corrosion. The charging pipelines examined after three years of service were completely intact.

We have found, however, that there was a rapid formation of pits in the pipes of unalloyed ductile iron, with a rapid decrease in the speed of corrosion after four years. This corrosion was not very serious, since very few (three or four) pipes needed to be replaced on four petroleum tankers thus equipped between 1951 and 1952. We believe, however, that the use of Ni alloyed ductile iron is a more sound practice.

#### Reply by F. L. LaQue:

We welcome the more extensive data on soil corrosion tests of ductile iron contributed by M. Paris of Pont-a-Mousson. This is additional evidence of the durability of ductile iron in underground applications.

Unfortunately, there are not enough data to measure precisely the contribution that nickel makes to the success of ductile iron in such tanker applications as heating coils. It may well be that the pipes that contain nickel will resist corrosion somewhat more than unalloyed iron. However, as noted by M. Paris, even the unalloyed iron performs quite satisfactorily, and the extent of its corrosion "is not very serious," since only three or four pipes have had to be replaced on four tankers in service for from six to seven years.

As mentioned in the paper, we can safely await the development of more practical experience to assess the advantage of having some nickel in ductile iron as used in tanker applications.

#### Comments by Sidney Low, Chapman Valve Manufacturing Co., Indian Orchard, Mass.:

The author is to be complimented for making these data on the comparative corrosion resistance of ductile iron available.

I am particularly interested in the comparisons of pearlitic and ferritic matrix ductile iron, since there is an increasing trend among consumers to require closer composition control along

**TABLE 2—Forgeson's Data for 1100 Alloy\* (Godard Discussion)**

Location of Specimen	4 YEARS			8 YEARS		
	Weight Loss, mpy	Pits (mils)		Weight Loss, mpy	Pits (mils)	
		Max.	Average Deepest 20		Max.	Average Deepest 20
Immersed.....	0.6	15	7	0.7	19	11
Half-tide.....	0.2	46	26	0.3	37	14

\* From Reference 4.

**TABLE 3—Weight Losses of Three Alloys At Three Exposure Sites (Godard Discussion)**

ALLOY	WT. LOSSES EXPRESSED AS mpy								
	Halifax, N.S.			Esquimalt, B.C.			Harbor Island N.C.		
	1 Yr.	2 Yr.	5 Yr.	1 Yr.	2 Yr.	5 Yr.	1 Yr.	2 Yr.	5 Yr.
1100 (2S).....	0.14	0.13	0.08	0.00	0.09	0.02	0.33	0.20	0.15
3003 (3S).....	0.00	0.13	0.07	0.00	0.00	0.04	0.30	0.24	0.14
5052 (67S).....	0.22	0.13	....	0.13	0.00	0.00	0.35	0.25	0.14

**TABLE 4—Pit Depth Data for Three Alloys At Three Exposure Sites (Godard Discussion)**

ALLOY	MAX. PIT DEPTH, MILS								
	Halifax, N.S.			Esquimalt, B.C.			Harbor Island N.C.		
	1 Yr.	2 Yr.	5 Yr.	1 Yr.	2 Yr.	5 Yr.	1 Yr.	2 Yr.	5 Yr.
1100 (2S).....	17	32	0	30	26	15	0	0	40
3003 (3S).....	13	15	21	5	20	0	0	0	13
5052 (67S).....	5	20	6	16	6	0	0	0	0

with carefully regulated ferritizing annealing treatments.

Mr. LaQue's data indicate, however, that in certain piping applications which involve velocity or turbulence sufficient to interfere with the corrosion product layer, ductile iron with a pearlitic matrix is preferred over the same iron annealed to produce principally a ferritic matrix structure.

Additional data would therefore be most valuable, particularly to us in the piping and fitting industry, relating the ferritic structure to exposure to more of the various corrosive media listed in the appendix.

Since the structure of a ductile iron matrix can be controlled by the chemical composition, and composition may be subject to restrictive specifications, a clear definition of the role of carbon, silicon, nickel and phosphorus in corrosion would also prove helpful.

I hope that the author plans to conduct further corrosion tests on ductile iron so that a compendium can be published relating composition, heat treatment, mechanical properties and corrosion resistance of ductile iron.

#### Reply by F. L. LaQue:

As pointed out in the paper, it is difficult to isolate the effects of individual elements, especially when external as well as internal factors will influence what happens. Pertinent data are continually being added to our records and can be made available to deal with specific questions as they arise or in published form as often as it is appropriate to provide them in this way. As ductile iron gets into more extensive use where corrosion is a factor, the choice of structure or composition will become dictated by practical experience. In the meantime the instances where such choice is likely to be critical, purely from the corrosion standpoint, are not likely to be numerous, and, in these cases, the general principles dealt with in the paper should provide a reasonably satisfactory guide to action.

#### Corrosion Rates in Port Hueneme Harbor by Carl V. Brouillette, Corrosion, Vol. 14, 352t-356t (1958) Aug.

Comment by Hugh P. Godard, Aluminum Laboratories Ltd., Kingston, Ontario, Canada.

This will comment on the paper "Corrosion Rates in Port Hueneme Harbor" by C. V. Brouillette.

Aluminum corrosion rates in the order of 1 mpy were found after one year while after 2.5 years the rates were about 0.5 mpy. Extensive and deep pitting also was reported.

The rates of pitting and of weight loss for aluminum alloys in Port Hueneme harbor are higher than will be found in most North American seawaters.

Mears and Brown<sup>1</sup> reported tensile property losses for several aluminum alloys and for steel in seven locations, but did not give pitting or weight loss data.

Thomas and Halferdahl<sup>2</sup> reported corrosion weight loss rates for several alloys at two locations. Their findings are given in Table 1. As can be seen, these rates are appreciably lower than those reported by Brouillette.

Walton and Engleheart<sup>3</sup> reported that 3003 and 5052 aluminum alloy pile casings at Pensacola Harbor, Florida, showed maximum pit depths of only 4 mils and 12 mils respectively after eight years in service. They further found that seawater in a flume at Kure Beach, N. C., produced an "average depth of attack" on several aluminum alloys of only 3 mils in six years. At Miami, Florida, 6061-T6 had a maximum pit depth of 3 mils in one year. These results also are much lower than found in Port Hueneme harbor.

More recently Forgeson et al.<sup>4</sup> reported results for 1100 alloy in the Pacific Ocean at Fort Amador, Canal Zone. These data are given in Table 2.

Panels of several aluminum alloys of 12 inch x 12 inch x 1/4 inch dimensions have been exposed by Aluminum Lab-



oratories Limited in three locations for periods up to five years. This information is reported in Table 3. Unfortunately, the original weights of the 1600 gram panels were taken to only 0.5 gram while after exposure the weights were taken to 0.01 gram. An error of 0.5 gram at the start could amount to  $\pm 0.08$  mpy at 1 year and  $\pm 0.02$  mpy at 5 years.

It is clear however that the rate of weight loss at Port Hueneme is several times that found at our three sites.

Pit depth data are given in Table 4. Despite the erratic behavior, which is typical of pitting data on a few specimens, the pit depths also indicate Port Hueneme to be more aggressive than our three sites since the maximum pit depth after two years shown above was only 32 mils compared to 84.5 mils reported by Brouillette. In addition, in our experience, the many pits shown on the specimen of alloy 1100 in Figure 1 and the localized corrosion of 3003 alloy in Figure 2 are not typical of the behavior of these alloys in seawater.

With regard to the tabulation and handling of pitting data, the value of "average pit depths" seems questionable. A true average would include many very shallow pits which would give a falsely low average value, and any other "average" is arbitrary and meaningless. Because pits are only important as they approach perforation the maximum depths are of the greatest interest. Statistical treatment of pitting data will give an even more reliable figure "the most probable maximum pit depth" as reported by Aziz.<sup>5</sup>

Our comment has been confined to the three aluminum alloys tested by Brouillette. We have many data on other aluminum alloys at the same three sites and in other locations that show similar weight loss and pitting rates. The fact that Brouillette found rates of 1-2 mpy at one year and about 0.5 mpy at 30 months indicates Port Hueneme to be

much more aggressive to aluminum than what we consider to be average ocean or harbor conditions.

#### References

1. R. B. Mears and R. H. Brown. Resistance of Aluminum Base Alloys to Marine Exposure. *Soc. of Nav. Archit. and Mar. Engrs.* (1944) Nov.
2. J. F. Thomas and A. C. Halferdahl. Corrosion Resistance of Some Commercial Aluminum Alloys in Seawater. *Can. Chem. and Process Ind.*, 28, 257-259 (1944).
3. C. J. Walton and E. T. Engleheart. Performance of Aluminum Alloys in Marine Environments. *Soc. of Nav. Archit. and Mar. Engrs.* (1949) Nov.
4. B. W. Forgeson, C. R. Southwell, A. L. Alexander, H. W. Mundt and L. J. Thompson. Corrosion of Metals in Tropical Environments. *Corrosion*, 14, No. 2, 73t-81t (1958) Feb.
5. P. M. Aziz. Application of the Statistical Theory of Extreme Values to the Analysis of Maximum Pit Depth Data for Aluminum. *Corrosion*, 12, 495t-506t (1956) Oct.

#### Reply by C. V. Brouillette:

The degree of corrosivity of the harbor water at Port Hueneme was judged by comparing the corrosion data on the 16 metals and alloys with that appearing in reference 1 for like material exposed to total immersion in sea water. Of course, the values reported in reference 1 vary from area to area for any particular material and, where possible, comparisons were made to West Coast data. Comparative data for metals and alloys other than aluminum appear in Table 5.

It is readily seen that the corrosion rates in Hueneme harbor for metals and alloys other than aluminum is equal to or lower than the corrosion rates given in reference 1.

The initial corrosion rates for 1100 aluminum in total immersion in Hueneme harbor, recalculated to average penetration from the total weight loss, at 18 months (0.7 mpy), 24 months (0.6 mpy) and 30 months (0.5 mpy) were almost identical to the rates reported by Forgeson et al (reference 4 of Dr. Godard's

TABLE 5—Corrosion Rates of Metals (Brouillette Reply)

METAL	Corrosion Rates in mpy at Port Hueneme	Corrosion Rates From Reference 1
Aluminum Bronze.....	0.2-0.7	1.2-1.9
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Monel.....	0.4-1.0	1.0
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Lead.....	0.1-0.2	0.4-0.6
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(Continued on Page 92)

oratories Limited in three locations for periods up to five years. This information is reported in Table 3. Unfortunately, the original weights of the 1600 gram panels were taken to only 0.5 gram while after exposure the weights were taken to 0.01 gram. An error of 0.5 gram at the start could amount to  $\pm 0.08$  mpy at 1 year and  $\pm 0.02$  mpy at 5 years.

It is clear however that the rate of weight loss at Port Hueneme is several times that found at our three sites.

Pit depth data are given in Table 4. Despite the erratic behavior, which is typical of pitting data on a few specimens, the pit depths also indicate Port Hueneme to be more aggressive than our three sites since the maximum pit depth after two years shown above was only 32 mils compared to 84.5 mils reported by Brouillette. In addition, in our experience, the many pits shown on the specimen of alloy 1100 in Figure 1 and the localized corrosion of 3003 alloy in Figure 2 are not typical of the behavior of these alloys in seawater.

With regard to the tabulation and handling of pitting data, the value of "average pit depths" seems questionable. A true average would include many very shallow pits which would give a falsely low average value, and any other "average" is arbitrary and meaningless. Because pits are only important as they approach perforation the maximum depths are of the greatest interest. Statistical treatment of pitting data will give an even more reliable figure "the most probable maximum pit depth" as reported by Aziz.<sup>5</sup>

Our comment has been confined to the three aluminum alloys tested by Brouillette. We have many data on other aluminum alloys at the same three sites and in other locations that show similar weight loss and pitting rates. The fact that Brouillette found rates of 1-2 mpy at one year and about 0.5 mpy at 30 months indicates Port Hueneme to be

much more aggressive to aluminum than what we consider to be average ocean or harbor conditions.

#### References

1. R. B. Mears and R. H. Brown. Resistance of Aluminum Base Alloys to Marine Exposure. *Soc. of Nav. Archit. and Mar. Engrs.* (1944) Nov.
2. J. F. Thomas and A. C. Halferdahl. Corrosion Resistance of Some Commercial Aluminium Alloys in Seawater. *Can. Chem. and Process Ind.*, 28, 257-259 (1944).
3. C. J. Walton and E. T. Engleheart. Performance of Aluminum Alloys in Marine Environments. *Soc. of Nav. Archit. and Mar. Engrs.*, (1949) Nov.
4. B. W. Forgeson, C. R. Southwell, A. L. Alexander, H. W. Mundt and L. J. Thompson. Corrosion of Metals in Tropical Environments. *Corrosion*, 14, No. 2, 73t-81t (1958) Feb.
5. P. M. Aziz. Application of the Statistical Theory of Extreme Values to the Analysis of Maximum Pit Depth Data for Aluminum. *Corrosion*, 12, 495t-506t (1956) Oct.

#### Reply by C. V. Brouillette:

The degree of corrosivity of the harbor water at Port Hueneme was judged by comparing the corrosion data on the 16 metals and alloys with that appearing in reference 1 for like material exposed to total immersion in sea water. Of course, the values reported in reference 1 vary from area to area for any particular material and, where possible, comparisons were made to West Coast data. Comparative data for metals and alloys other than aluminum appear in Table 5.

It is readily seen that the corrosion rates in Hueneme harbor for metals and alloys other than aluminum is equal to or lower than the corrosion rates given in reference 1.

The initial corrosion rates for 1100 aluminum in total immersion in Hueneme harbor, recalculated to average penetration from the total weight loss, at 18 months (0.7 mpy), 24 months (0.6 mpy) and 30 months (0.5 mpy) were almost identical to the rates reported by Forgeson et al (reference 4 of Dr. Godard's

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(Continued on Page 92)

## Electrolysis Committee Needs Information

Information about electrolysis (corrosion coordinating) committees not affiliated with NACE T-7 is needed to increase the committee's effectiveness. C. A. Erickson, Jr., People's Natural Gas Co., 2 Gateway Center, Pittsburgh 22, chairman of T-7, seeks the names of committees and their officials so that he can contact them concerning T-7's activities.

Mr. Erickson says there is no known directory of these committees. He believes a compiled list of active committees will be helpful to all of them. Those who know of committees not on the T-7 list (See CORROSION, October, Page 12) are urged to send their names to Mr. Erickson.

## Rocket Engine Coatings May Have Industrial Uses

Flame deposited coatings developed in connection with rocket engines are believed to have important industrial applications. These coatings, which can be made of a variety of metals, ceramics or combinations of metals and ceramics, are designed to protect the base metal on which they are deposited from the effects of high temperature gases containing corrosive components moving at high velocities.

Developments in this field were the principal topics of discussion at a meeting of T-5B on High Temperature Corrosion at Cincinnati during the October meeting of North Central Region. The meeting was one of seven scheduled meetings of technical committees.

Among the characteristics of the coatings which gives them unusual stability, it was pointed out, was the high densities achieved, in some instances as high as 98 percent of theoretical maximum density. Another important characteristic is that the method used permits application of materials as coatings which have a high melting point themselves, sometimes in the ranged 1500-2000 F.

### Method of Deposit Described

The method being used experimentally to deposit the coatings involves the use of a rapidly moving air stream heated to temperatures in the 1500 F range. Coatings to be deposited are introduced into the stream as powders.

The piece to be coated is placed in the air stream so that the molten powder will impinge on it and coalesce into a coating. Examples were shown of homogeneous coatings in excess of 1/4-inch in thickness deposited on a variety of base materials.

Some of the materials being deposited in this manner are metallic, others ceramic, including aluminum oxide and similar compositions and various metallic-ceramic composites. Workers familiar with the process believe that when techniques have been further developed these coatings will have important industrial applications in the high temperature field and where coatings of almost 100 percent density are useful.

## Problems of Fuels Containing Boron Discussed By Committee on High Temperature Corrosion

Problems involved in the use of high energy fuels containing boron were discussed at a meeting of T-5B on High Temperature Corrosion at Cincinnati during the North Central Region meeting in October. The analogy between the effect of boron on some materials and the effect of vanadium pentoxide was pointed out. High energy fuels containing boron are being tested for use in rockets and jet engines.

Many of the discoveries in connection with this problem have developed from research projects sponsored by the Air Force at laboratories of industrial companies and universities. Recently developed coatings have extended the life of some metals from minutes to about 150 hours, it was pointed out.

### Boron Forms Eutectic

The mechanism postulated for the attack by boron compounds on materials includes diffusion into the material, as in nickel where a eutectic of lower melting point than the nickel is formed, as in aluminum or titanium alloy where boron dioxide causes the formation of free boron, which produces a like eutectic and in iron-cobalt alloys in which it dissolves the protective oxides.

Several materials, however, have shown satisfactory resistance to boron compounds, among them being Hastelloy X and Inconel. Dayton University has

## Storage Tanks—

(Continued From Page 91)

committee was useful in dealing with an organization like NATO.

A change in the method of installing coupons in a pipe line was discussed by the chairman. The coupon is now rigidly mounted to prevent swinging.

The value of information obtained from coupons was stressed by another guest. Insufficient inhibitor protection can be indicated by coupon tests, he said. Often inhibitor manufacturers profit from information received from coupon tests which indicate insufficient inhibiting action.

Various methods of running rust tests were discussed by the committee. The committee agreed to collect rust tests methods, with the intent of making these tests uniform.

## NEW COMMITTEE WORK PROPOSED ON ALUMINUM

Formation of a unit committee under T-2 "Pipe Line Corrosion" has been suggested to explore the possibility of cathodic protection of buried aluminum pipe lines. This problem has been relatively unexplored.

Persons desiring to participate in this committee should write to: Dr. H. P. Godard, Aluminium Laboratories, Ltd., P. O. Box 84, Kingston, Ontario, Canada.

An organizational committee meeting will be held in Chicago next March if sufficient interest is shown.

conducted exhaustive tests on some 30 materials in the 1500 F range.

### Protection Against Vanadium

It also was brought out that additives to Bunker C oil have beneficial effect in reducing attack by vanadium pentoxide with sodium residue on boiler parts. Magnesium gives good protection while aluminum did not protect, according to one speaker. On the other hand mixtures of magnesium and aluminum not only gave good protection but did so without creating objectional deposits.

A discussion on high temperature effects on hydrogen sulfide developed the information that at 1500 F all hydrogen sulfide was converted to sulfur dioxide while in the range 900-1000 F about one-half was converted. Inconel is useful for its catalytic effect in reducing hydrogen sulfide to sulfur dioxide.

One speaker pointed out that the ASME report on combustion gas would be issued soon.

## Coated Tubing—

(Continued From Page 91)

One operator reported the application of the so-called short squeeze or tubing displacement treatment in a gas lift well. He indicated that 99 percent protection was obtained in the well for six months. This method of inhibition appeared promising, but its merits compared to the squeeze method are to be studied further. Much interest was shown in the squeeze method, and it will be discussed at future meetings.

An average of four to six short courses on corrosion control are held each year in the United States with the cooperation of NACE.



## TECHNICAL REPORTS

on

## MARINE COATINGS

**T-1M** Suggested Coating Specifications for Hot Application of Coal Tar Enamel for Marine Environment. A Report of NACE Technical Unit Committee T-1M on Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations. Publication No. 57-8. Per Copy \$5.00.

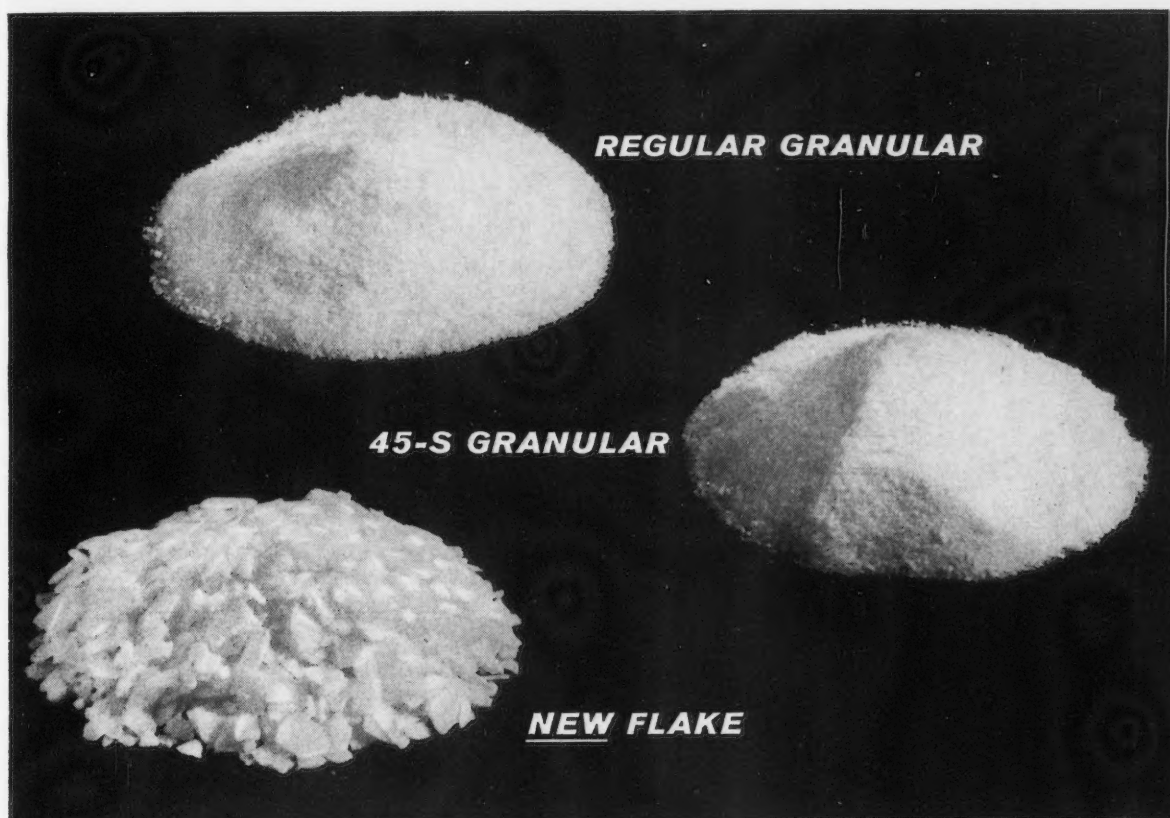
**T-1M** Suggested Painting Specifications for Marine Coatings. A Report of NACE Technical Unit Committee T-1M on Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations. Publication No. 57-7. Per Copy \$5.00.

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## NACE NEWS

### Houston Short Course Tentative Program Set For Jan. 22-23

Tentative subjects and speakers have been announced for the short course in fundamentals of corrosion control set for January 22 and 23. Sponsored by the Houston Section, the short course will be held at Oberholtzer Hall, University of Houston.

Nine subjects and speakers have been scheduled:

1. Fundamentals of Corrosion Processes by C. A. Woody, United Gas Corp.
2. Underground Corrosion and fundamentals of Cathodic Protection by M. A. Riordan, Rio Engineering Co.
3. External Coating for Underground Protection by W. R. Curley, Royston Laboratories.
4. Internal Corrosion: Corrosion Inside of Pipe Lines and Tankage and Use of Inhibitors for Corrosion Control. Speakers to be announced.
5. Designing for Corrosion Control of Pipe Lines and Corrosion Consideration in Pipe Line Design by Pierce R. Butterfield, World Supply Co.
6. Instruments for Corrosion Measurement. Speaker to be announced.
7. Control of Atmospheric Corrosion and Surface Preparation and Painting by John Weis, Diamond Alkali Co.
8. Installation and Operation of Rectifiers. Speaker to be announced.
9. Galvanic Anodes for Cathodic Protection. Speaker to be announced.

Directed toward practical applications of corrosion control, the short course has been designed for operating field men such as junior engineers, foremen, electricians and technicians. Any person interested in corrosion is invited.

Registration fee is \$10 per person. No meals are included in the fee. Reservations for registration should be sent to William Campbell, F. H. Maloney Company, 2301 Texas Ave., Houston 2.

Registration will begin in Oberholtzer Hall at 7:30 am, January 22.

The first session will be an introduction to basic fundamentals of corrosion with all registrants attending. Remaining classes will be divided into small groups.

Hotel accommodations should be handled by the individual.

### North Texas Hears Talk on Role of Metal in Corrosion

The Role of Metal in Corrosion was the subject of the technical program for the North Texas Section meeting November 3. Archie Broodo, speaker, is head of a component development branch at Texas Instruments, Inc., Dallas, where he works with metals, alloys and their surfaces. In his talk he discussed recent developments in surface theory pertaining to corrosion phenomena.



HOUSTON SECTION heard Troy R. Stilley, Good-All Electric Mfg., Co., Ogallala, Neb., speak on the new developments in cathodic protection rectifiers. At left is E. P. Doremus, a section member.

### Selenium Stack Use Described as New Rectifier Development

A selenium stack added to a bond to combat stray current corrosion in underground structures will not only provide the required protection but in many cases will also use stray a-c current to provide protection. This phenomenon was explained by Troy Stilley, speaker at the Houston Section meeting, October 14. He is general manager, electrical-mechanical division, Good-All Electric Mfg., Co., Ogallala, Neb.

Mr. Stilley gave an example of a section of pipe line picking up 100 amperes of stray current which was to be bonded to a negative buss on a trolley system so that the pipe-to-soil potential would not change over one-tenth of a volt. By ohms law, the resistance of the bond would have to be 1 one-thousandth of an ohm. The resistance of a selenium stack inserted in the bond would be approximately 2 one-thousandths of an ohm. The assumption under these conditions is that the shift in pipe-to-soil potential would be enough that stray current corrosion would still exist.

A selenium stack has been used in several applications of this type. When the bond with the stack was added, the potential of the pipe line was always shifted in the protected direction instead of in the direction which indicates stray current corrosion. In most cases, there was enough stray a-c current in the ground to supply power which would be rectified by the selenium stacks and actually result in a free power source for cathodic protection, Mr. Stilley said.

In discussing meter switches, Mr. Stilley said that much of the variation obtained in meter readings was caused by changing resistance in the meter switch from corrosion on the switch contacts. This problem could be solved by using a circuit in which the ammeter is shorted out when not in use and the contacts are open to allow the ammeter to read when the button is pushed. The

(Continued on Page 96)

### Pipe Line Short Course Scheduled at Tulsa

The Tulsa Section's tenth annual Pipe Line Short Course will be held February 11-13 in the Mayo Hotel. Emphasis will be on the role of operating personnel in corrosion control. Specific examples will be related to lectures and discussions.

The regular section meeting will be held January 26 at the Mayo Hotel. No meeting was scheduled in December. A trip through installations of the Phillips Petroleum Co., Bartlesville, Okla., was planned for the November meeting.

Dynamometer well studies to minimize corrosion fatigue of rods and tubing was discussed by Douglas O. Johnson for the October meeting.

### More Booths Sold for 1959 Corrosion Show

A total of 77 exhibitors have purchased space for the 1959 Corrosion Show to be held March 17-19 during the 15th Annual NACE Conference in Chicago.

Over 100 booths have been sold for the show.

Companies to have exhibits not included in the list published in the October CORROSION are the following:

Alloy Steel Products Company  
Calag Chemical Company  
Cathodic Protection Service  
Corning Glass Works  
Cosasco Division, Perfect Circle Corp.  
The DeVilbiss Company  
General American Transportation Corp.  
The Glidden Company  
Gulf States Asphalt Company  
M. C. Miller Company  
Oregon Metallurgical Corp.  
Phelps Dodge Copper Products Corp.  
Plicoflex, Inc.  
Polyken Sales Division, Kendall Company  
Radio Receptor Company  
Remco Mfg. Company  
Wayte Industrial Supply Corp.

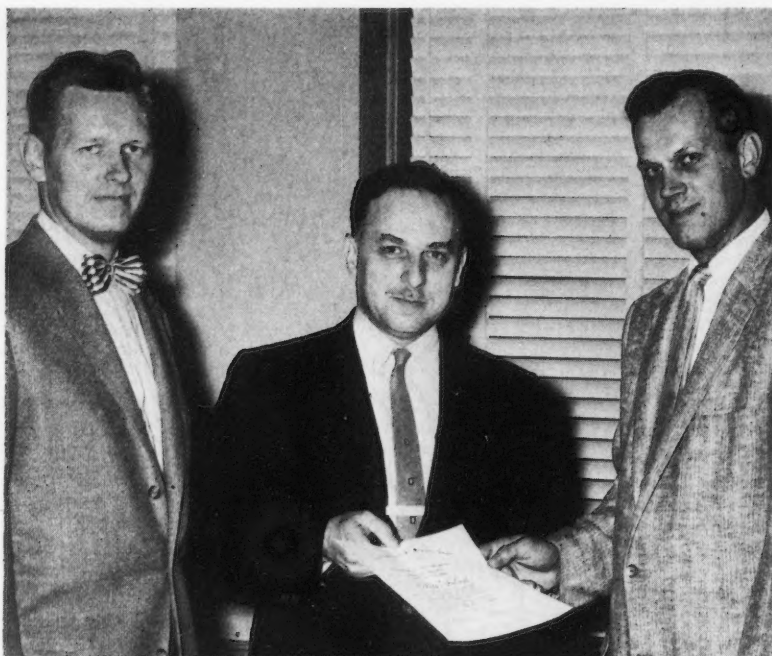
### 15th Annual Conference Plans Being Completed

Plans for the 15th Annual Conference in Chicago March 16-20 are being completed.

Some symposia programs are complete and others are filling rapidly, according to program chairmen.

Pre-conference round-up information is being prepared by the Central Office for publication in the January issue of CORROSION. Titles and abstracts of papers with authors' names and biographies will be given. Business and social items will be listed also.

Over 60 technical committee meetings are being planned. A meeting of the Inter Society Corrosion Committee is also scheduled.



FIRST PRESENTATION of the L. David Cook Memorial Award is being presented to McKay E. Padmos (right), 1958 metallurgical engineering graduate of Wayne State University. Presentation was made at a recent meeting of the Detroit Section by R. K. Swandby, program chairman (left), and L. W. Gleekman, vice chairman (center). The award was established by an initial grant of \$1000 from Mrs. Cook and the Detroit Section to the Department of Chemical and Metallurgical Engineering at Wayne State.

## Panel Held at Philadelphia On Underground Corrosion

A panel discussion on underground corrosion with questions from the audience was the technical program for the Philadelphia Section October meeting. With the meeting announcement, a post card was mailed to members requesting their underground corrosion problems for discussion at the meeting.

Panel members and their subjects

were H. L. Hamilton, president, A. V. Smith Engineering Co., on Cathodic Protection; C. F. Pogacar, chief metallurgist, Nice Ball Bearing Co., on Materials of Construction and N. T. Shideler, protective coatings research manager, Pittsburgh Coke and Chemical Co., on Protective Coatings.

An average of four to six short courses on corrosion control are held each year in the United States with the cooperation of NACE.

## NACE's 58th Section Formed at Wilmington

The 58th NACE section was formed in October with the first preliminary meeting of the Wilmington Section in the Northeast Region. Thomas F. Degnan was elected temporary chairman and James M. Brady temporary secretary.

The new section was approved by the Northeast Region Board of Trustees on May 5.

Area to be covered by the section includes parts of four states: New Castle County in Delaware; Cecil County in Maryland; Salem, Cape May and Cumberland Counties in New Jersey and Lancaster County and the southern half of Delaware County in Pennsylvania.

## New York Section Hears Microbe's Role in Corrosion

Underground Corrosion by Microbiological Action, by F. E. Kulman, Consolidated Edison Co., New York, was the technical subject scheduled for the October Metropolitan New York Section meeting.

Mr. Kulman is in charge of corrosion design for his firm's underground plant, including pipe type, lead sheath cables and gas and steam piping systems. A member of NACE since 1946, he is serving on several committees and is chairman of the Utilities Industry symposium for the 15th Annual Conference and 1959 Corrosion Show.

## Genesee Valley Section

Discussion on General Corrosion Problems was the technical subject scheduled for the October meeting of the Genesee Valley Section. The meeting was held in Rochester, N. Y. Members with deterioration problems in coatings, fibers or metals were invited to present them for discussion.

## Atlanta Officers Elected

New officers for the Atlanta Section have been elected. They are Chairman C. Jay Steele, Steele Associates; Vice Chairman James P. McArdle, Jr., American Telephone and Telegraph; and Secretary-Treasurer I. N. Howell, Southern Bell Company.

## Selenium Stacks—

(Continued From Page 95)

ammeter will read accurately when the button is pushed because there are no switch contacts in the circuit.

Cost of constant current control equipment has dropped drastically, Mr. Stillely said. A unit costing \$2500 in 1954 would cost approximately \$450 today.

In recent years, high voltage silicon diodes and higher voltage selenium plates have caused rectifier designers to change their approach to circuits and designs, according to Mr. Stillely.

Northeast Region Meeting will be held October 5-7, 1959, at Baltimore, Maryland.

North Central Region Meeting will be held in Cleveland, October 20-22.

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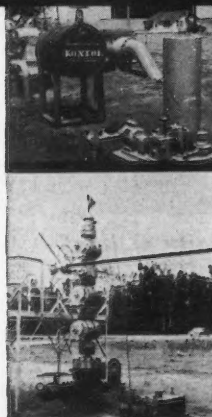
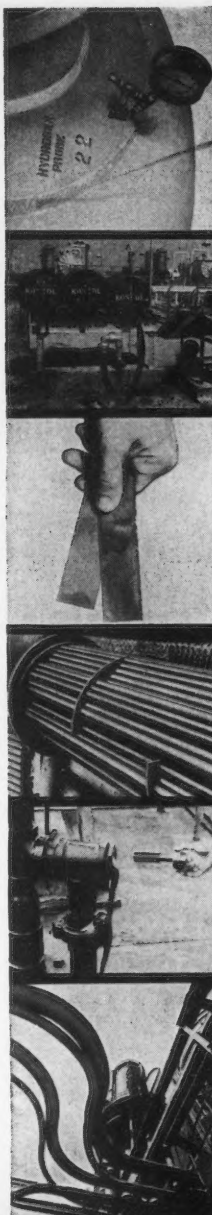
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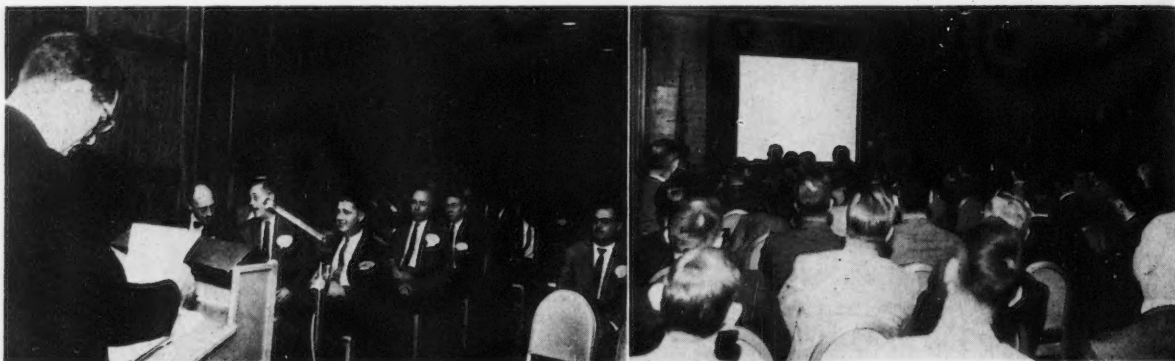
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AT NORTH CENTRAL REGION'S October 15-17 in Cincinnati Sylvan B. Falck (upper left), Inner-Tank Lining Corp., opens the coatings symposium; upper right, Mars G. Fontana, Ohio State University, presents his paper on stress corrosion cracking of stainless steels. Left, persons attending the Fellowship Hour and buffet at the Sheraton Gibson Hotel.



## Symposia Sessions Have Capacity Attendance At North Central Region Meeting in Cincinnati

Capacity attendance at technical symposia sessions featured the October 15-17 meeting in Cincinnati of North Central Region. Registration was 201. The majority of those registered attended technical sessions and committee meetings.

Comments were uniformly favorable on the technical content of the program, the smooth operation of sessions and the exceptional interest in the event shown by registrants, the majority of

whom were from places other than Cincinnati.

Especially enjoyable were Fellowship Hour and buffet on Thursday evening. These were attended by wives of visiting registrants and by many of the wives of Cincinnati members.

The meeting also gave an opportunity for the Cleveland Section members who will have charge of the 1959 regional program to discuss program arrangements.



GUESTS AT CINCINNATI during the meeting of North Central Region were Mrs. J. R. Hagerman and Mrs. L. O. Risetter of Chicago who are members of the entertainment committee for the NACE 15th Annual Conference to be held at the Sherman Hotel next March.

## Region Announces '59-'62 Meetings

Places have been set for meetings of North Central Region through 1962. The places were selected at a meeting of the region's officers present at the October Cincinnati session over which W. E. Hare, Hare Equipment Co., Cincinnati, presided.

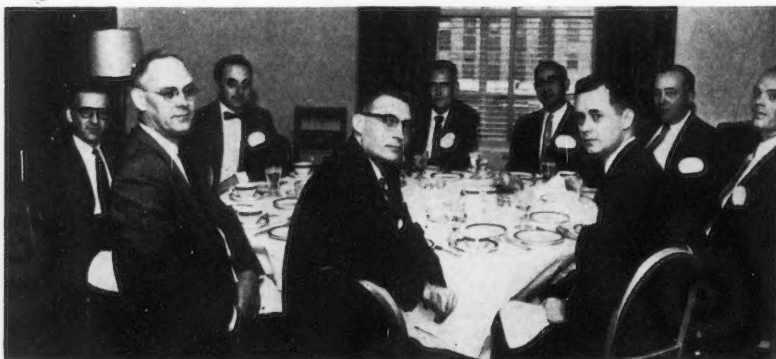
The schedule adopted was as follows:

- 1959—Cleveland.
- 1960—Milwaukee.
- 1961—St. Louis.
- 1962—Kansas City.

The 1961 and 1962 dates were considered tentative because of a possible conflict with the March, 1962, meeting of NACE at Kansas City.

The discussion also involved methods of financing regional meetings, election of trustees by sections and a change in regional boundaries. Some anomalies in connection with the existing boundaries of regions were considered.

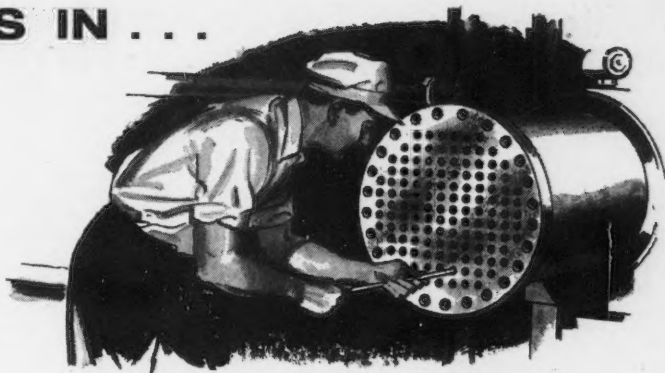
Urgent need for a written guide for use of those conducting regional meetings was expressed.



PLANS FOR THE 1959 meeting in Cleveland of North Central Region were discussed at this meeting of region officers at Cincinnati during the October 15-17 conference there. Seated at the table beginning at the center foreground and going clockwise: A. O. Fisher, Monsanto Chemical Co., St. Louis, St. Louis Section treasurer; L. C. Wasson, A. O. Smith Corp., Milwaukee, NACE director; A. McConnell, Geo. J. Meyer Mfg Co., Cudahy, Wis., regional treasurer; L. W. Gleekman, Wyandotte Chemical Corp., Wyandotte, Mich.; C. W. Ambler, Jr., American Zinc, Lead & Smelting Corp., East St. Louis, Ill., St. Louis Section chairman; B. Husock, Harco Corp., Cleveland; W. E. Hare, Hare Equipment Co., Cincinnati, regional chairman; F. V. Vanderhenst, Jr., NACE Central Office; R. C. Weast, Case Institute of Technology, Cleveland, Ohio.

## WATER PROBLEMS IN . . .

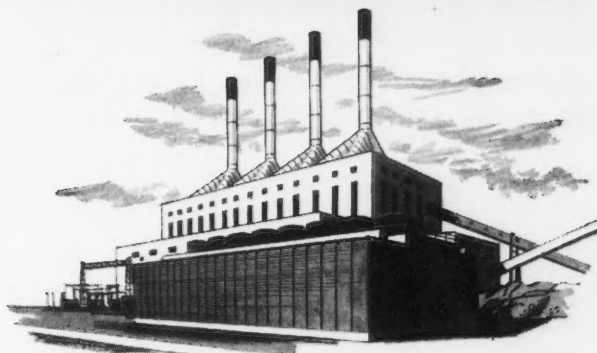
heat exchangers ?



pipe lines ?



cooling towers ?



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## Publication Committee Discusses Problems At South Central Region Meeting in October

Numerous problems of NACE in the publications field were discussed at the October 20 meeting of the Publication Committee at New Orleans during the South Central Region meeting. It marked the first committee meeting held in recent years at other than a national NACE meeting. R. S. Treseder, Shell Development Co., Emeryville, Cal., presided. Among those attending were Ivy M. Parker, Plantation Pipe Line Co., Atlanta, editor of CORROSION; J. E. Loeffler, Thornhill-Craver Co., Houston, advertising subcommittee chairman; R.

D. Misch, Argonne National Laboratory, Lemont, Ill., chairman of the abstract subcommittee; and Fred M. Reinhart, National Bureau of Standards, Washington, D. C., chairman of the editorial review subcommittee.

Also present were members of the NACE Executive Committee and NACE Central Office staff.

Budget matters, means of improving publications for members and such matters as policy on microfilming back issue files were considered. Considerable time was spent considering future plans for development of CORROSION.



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## Technical Section to Have Larger Type

Size of type for the Technical Section of CORROSION will be changed beginning with the January issue to a nine point Baskerville type on a nine point line.

During 1958, the section was printed in an eight point type on an eight point line.

The three-column format will remain unchanged.

## Region Manual Issued For Planning Meetings

A manual to guide North Central Region conference officials in planning and organizing the 1959 region conference has been distributed to chairmen of the conference committees. The conference will be held in Cleveland at the Statler Hilton Hotel, October 20-22.

The indexed manual has a section for each committee which states what the committee is to do and the dates on which its tasks are to be completed.

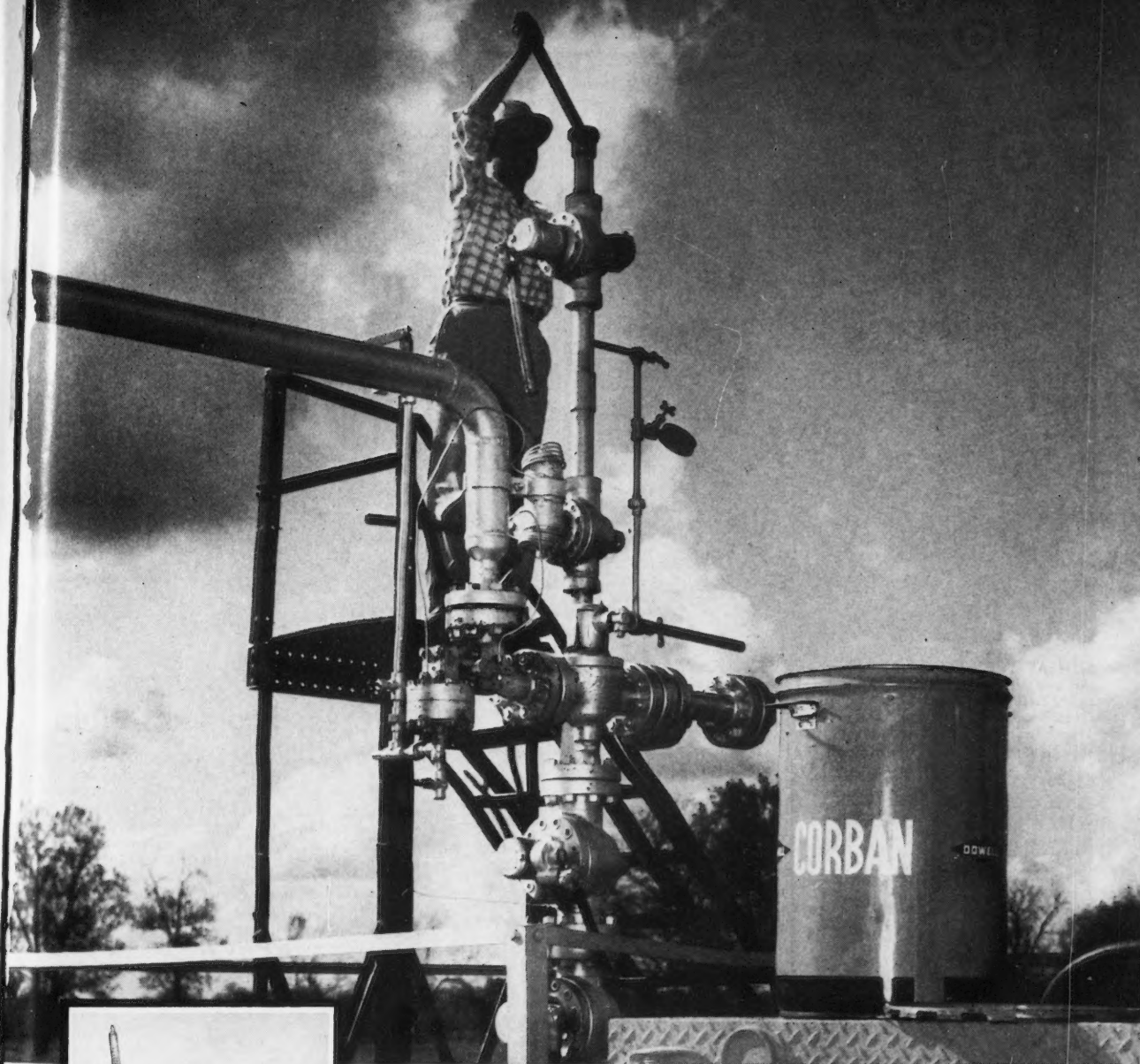
In addition to being responsible for his own committee, each chairman is asked to contribute his suggestions and opinions on all phases and problems of the conference.

A general planning committee, composed of the chairman of the various committees, the conference general chairman and vice chairman will coordinate the activities of the working committees. This general planning committee will meet each month. The first meeting was held August 27.

The committees and their chairmen are: Lester D. Mills, Jr., The Standard Oil Company of Ohio, general chairman; Alva Corlett, Jr., Harco Corp., vice-chairman; Edward W. Vereeke, Heil Process Equipment Corp., technical program chairman; John Scott, Truscon Labs., finance chairman; Richard W. Mohler, Koppers Co., Inc., Tar Products Div., hotel arrangements chairman; Austin K. Long, Glidden Co., registration and printing chairman; Robert Schantz, Sherwin-Williams Co., publicity chairman; Thomas S. Howald, Chase Brass & Copper Co., Inc., fellowship hour chairman; and Harry W. Hesford, Jr., Harco Corp., plant trips chairman. The Cleveland Section officers will comprise the reception committee.

Plans have been made to invite the NACE technical committees to meet during the conference, and preliminary work on this was scheduled for the November 18 meeting of the general planning committee. The November meeting also reviewed committee progress, financial requirements of all committees, and a review by the hotel arrangements committee of attendance reports at past regional meetings. For the December 18 agenda, the financial committee will present a preliminary budget. A review of over-all committee progress will also be made then.

The 15th Annual NACE Conference and 1959 Corrosion Show will be held March 16-20 at the Sherman Hotel, Chicago.



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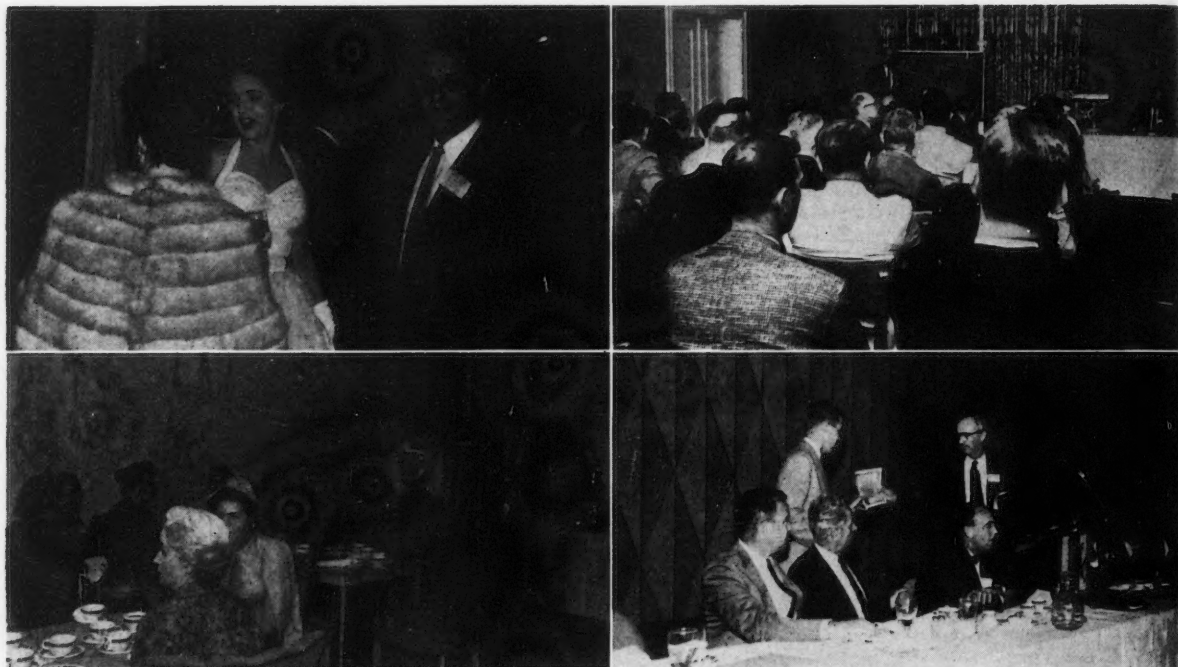
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SEEN AT THE NEW ORLEANS meeting of South Central Region October 20-24: Upper left, Mr. and Mrs. J. C. Spalding, Jr., Sun Oil Co., Dallas. Mr. Spalding is regional chairman. Upper right, P. D. Muir, Dowell, Inc., Tulsa chairman, addresses a meeting of T-1 D on Sour Oil Well Corrosion. Lower left, some of the ladies enjoying morning coffee. Lower right, J. E. Loeffler, Thornhill-Craver Co., Houston, chairman of the region's educational committee presents the region's certificate of achievement to Luedric D. Harman, Ponca City, Okla., high school senior. Mr. Harman also received a cash award of \$150.



THE EXHIBITION held concurrently with South Central Region's Oct. 20-24 conference at New Orleans was well attended. This scene was taken during the "free afternoon" arranged to give all registrants an opportunity to see the exhibit.

## Attendance Good at South Central Region

Attendance at technical committee meetings and symposia during South Central Region's Conference in New Orleans October 20-24 was sustained at a high level throughout the five days. This meeting was the first held by the region in which the program of technical committee meetings was separated chronologically from the technical symposia. Attendance at the half-day session on the final day was good in both symposia.

High interest was shown in the concurrent exhibition which was open during the middle three days of the session.

As usual the "free" half day developed the largest crowds. Attendance during the remainder of the show was good also.

As expected, New Orleans proved to be a major attraction for the ladies, with 138 registered for the various social events. Total registration was 776.

Attendance at technical committee meetings was good generally with the largest number representing the petroleum industry. This was expected in view of the region's orientation in the petroleum industry area.

## Scientific Education Needs Engineers' Help

Engineers were urged to make a personal contribution to the education of the nation's youth in science at the October meeting of the South Central Educational Committee at New Orleans. Personal sacrifice by men in scientific pursuits will be necessary if the nation is to keep abreast of the rest of the world in science education, the committee was told.

The meeting, attended by members of the committee representing the several states of the region and by regional officers and members of NACE Central Office staff, was presided over by J. E. Loeffler, Thornhill-Craver Co., Houston, chairman. Next meeting of the committee will be during the region's 1959 meeting at Denver.

### Activity Is Reviewed

Mr. Loeffler reviewed the committee's work so far and outlined some of the activities scheduled during the next year. He noted that in recent months North Texas Section has named Edwin H. Muelhouse, Lone Star Gas Co., Dallas, as chairman of its education committee.

L. W. Hartkemeier, Colorado School of Mines, Golden, was named the committee's liaison representative with the Future Science Clubs of America, the organization to be fostered by the federal government among high school students studying science.

Mr. Loeffler expressed hope that every section in South Central Region would name an education committee to work with schools in furtherance of science education.

### Houston Cooperation Cited

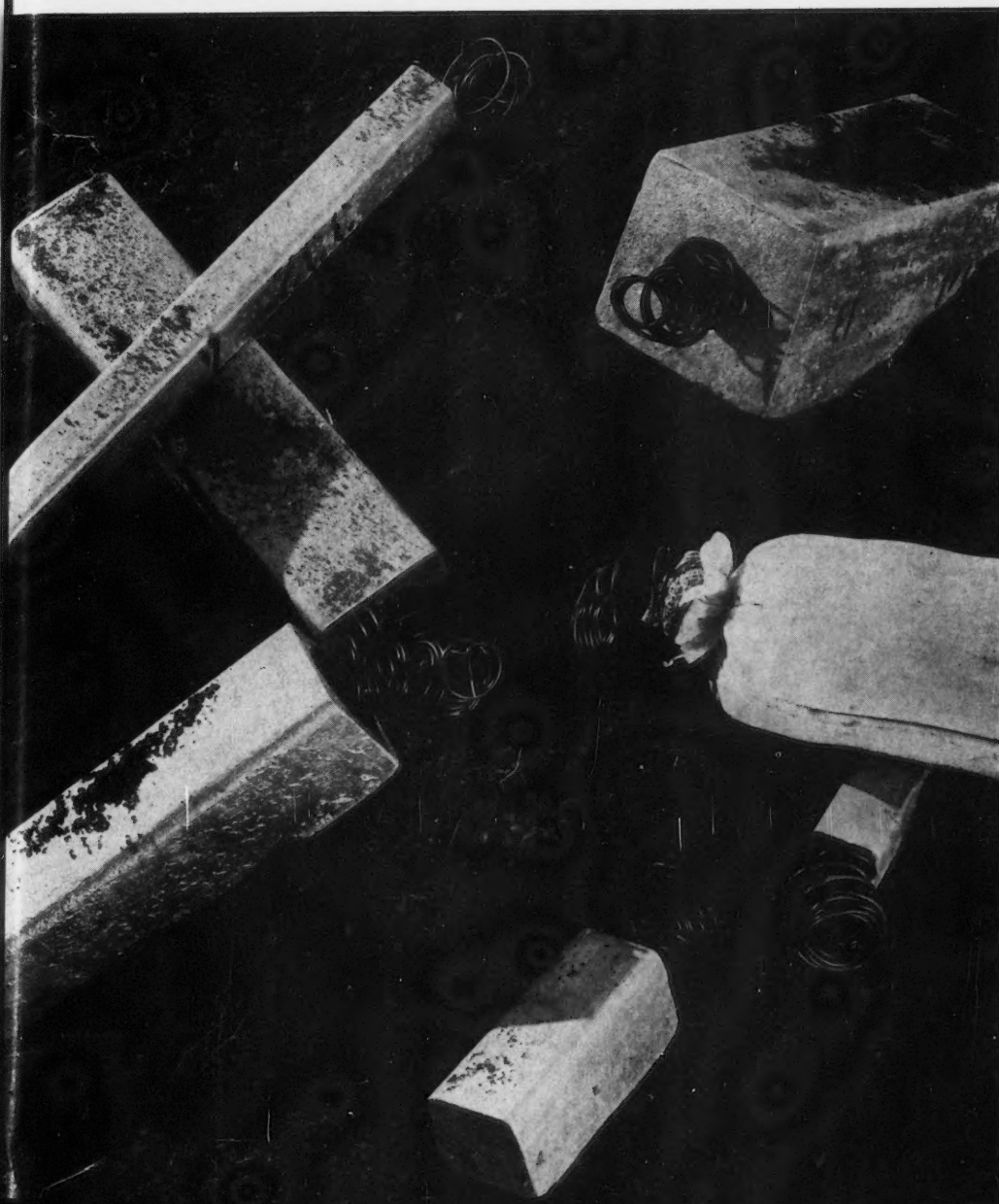
The manner in which Houston Section is working through the Houston Scientific and Technical Society to assist

(Continued on Page 104)



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## Canadian Region Eastern Meeting to Be Jan. 12-14 in Montreal

Emphasis will be on practical discussion of corrosion work at the eastern meeting of the Canadian Region to be held January 12-14 at the Sheraton Mount Royal Hotel in Montreal. Discussions will be concerned with pulp and paper manufacture, mining and metallurgy, chemical processing, oil refining and petrochemical plants.

Highlights of the technical meetings will be the following papers: Corrosion Research in the Pulp and Paper Industry, by R. B. Kesler, Institute of Paper Chemistry, Appleton, Wis., Corrosion in Alkaline Pulping Digesters, by E. W. Hopper, and Corrosion Problems in Smelting and Refining, by G. S. Farnham, International Nickel Co.

Other papers to be presented are High Temperature Corrosion, by H. G. Bennett, British American Oil Co., Corrosion Problems Encountered in Chlorine Processing Units, by N. Zundel, Vickers Krebs, Corrosion Problems in Cooling Water Systems in Eastern Canada, by J. B. Hambley, Alchem, Ltd., and Some Notes on Cathodic Protection in Tidal Estuaries, by D. B. Bird and H. G. Burbidge, Aluminum Company of Canada, Ltd.

## Portland Section Meets

Atmospheric Corrosion was the subject for the November 13 meeting of the Portland Section. Title of the technical program was How to Save a Million Dollars. A 20-minute color film was also shown.

The Western Region Conference is scheduled for September 29-30, October 1, 1959, at Bakersfield, Cal.



## SECTION CALENDAR

### Dec.

- 2 Shreveport Section. Caddo Hotel. Fellowship hour, 6 p.m. Student Education Night.
- 8 Kansas City Section
- 10 Metropolitan New York Section. In New York City. Corrosion of Iron and Steel, by C. P. Larrabee, United States Steel Corp., Monroeville, Pa.
- 12 Birmingham Section. Cathodic Protection Project for Large Housing Developments, by Jay Steele, Steele & Associates, Inc.
- 12 San Diego Section.
- 16 Cleveland Section. Cleveland Engineering and Scientific Center. Stainless Steel, by W. Luce.
- 18 Vancouver Section.

### Jan.

- 6 Shreveport Section. Caddo Hotel. Fellowship hour, 6 p.m. Offshore Corrosion Control.
- 14 Metropolitan New York Section. In Newark. Use of Epoxy Resins in Resistant Coatings, by H. W. Howard.
- 15 Vancouver Section. In B.C. Electric Bldg. Auditorium. Aluminum in Marine Environments, by T. J. Summerson, Kaiser Aluminum.
- 20 Chicago Section. Bacteria's Role in the Corrosion of Iron, by D. M. Updegraff, Minnesota Mining & Mfg., Co.
- 20 Cleveland Section. Cleveland Engineering and Scientific Center. Joint meeting with Electrochemical Society.
- 22 Kanawha Valley Section.
- 26 Tulsa Section. Mayo Hotel.
- 26 Atlanta Section.
- 27 Panhandle Section.

## 1959 Officers Elected For Southeast Region

Two days of technical symposia and election of 1959 officers were held at the October meeting of the Southeast Region in Richmond, Va.

Past Chairman's Certificates were presented to the following: E. B. Ayers, James T. MacKensie, E. C. Range, Robert A. Davis, E. P. Tait, John B. West, Herbert C. Van Nieuhuys, J. Frank Putnam, Fred D. Stull, Arthur B. Smith and Joseph Frink.

New officers elected were Chairman Robert D. Williams, Celanese Corp., Charlotte, N. C., Vice Chairman George M. Jeffares, Plantation Pipe Line Co., Atlanta, Ga., Secretary-Treasurer John B. Paisley, Jr., American Telephone and Telegraph Co., Birmingham, and Assistant Secretary-Treasurer John B. West, Aluminum Company of America, Atlanta, Ga.

The 1959 meeting of the region has been planned for October 1-2 at Jacksonville, Fla.

## Plastic Engineers Society To Hold Meeting Jan. 27-30

Topics of probable interest to NACE members will be presented at the annual technical conference of the Society of Plastic Engineers to be held January 27-30 in New York City.

One session will be on test methods with papers to be given on Determining Hardness and Stress Cracking and Methods of Measuring Flammability of Plastics.

Another session will be devoted to Ultra-High Temperature Reinforced Plastics. Papers to be included are Behavior of Reinforced Plastics in Contact With Hot Gases, Application of Resins to Re-entry Ablation and Designing Reinforced Plastic Systems for Ultra High Temperature Use.

A session on Permanence Properties will include a paper entitled Comparison of Short-time Versus Long-time Properties of Plastic Pipe Under Hydrostatic Pressure.

## Scientific Education—

(Continued From Page 102)

science teachers in the schools was explained by M. A. Riordan, Rio Engineering, Houston, Texas representative on the committee. Mr. Riordan told of work with two schools in the Houston area during the past year.

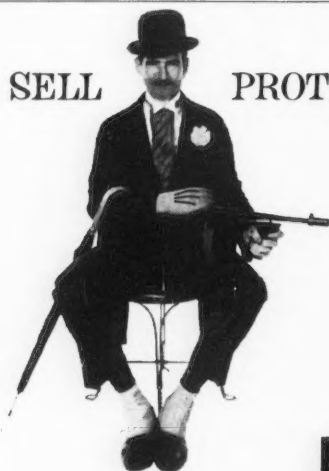
Mr. Riordan also distributed a working draft of a proposed elementary corrosion manual. He suggested that those interested in the project send additions, changes and revisions to him.

While this publication will be the work, principally, of South Central Region, ultimate distribution probably will be under guidance of the NACE Publication Committee, it was pointed out.

### Short Course Manual

Charles L. Woody, United Gas Corp., Houston, is preparing a manual outlining the procedure whereby corrosion control short courses may be conducted by sections. This publication is believed to be of immediate importance to many NACE sections which are active in short course promotion.

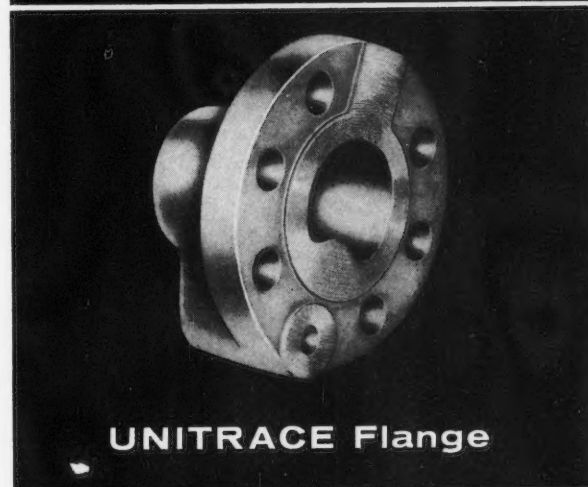
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of steam-traced piping*

Users of steam-traced pipe have found that valuable savings in cost plus greatly improved efficiency are automatic when they use ALCOA® UNITRACE. It provides steam and product passages extruded in a single unit of light, strong, corrosion-resistant aluminum alloy... completely eliminating the need for costly external steam jackets or inefficient tracer tubes. Now a new flange and trace cap provide fittings and connections designed for UNITRACE which are compatible with conventional piping. You can now design completely integrated UNITRACE piping systems and get all these benefits:

### **Low heat loss . . . high internal heat transfer**

The recently introduced *round* configuration of UNITRACE makes possible most efficient internal heat transfer with minimum external radiation loss.

### **Easy, economical joints and connections**

The special UNITRACE Flange mates with all 150-lb ASA flanged connections, permits use of standard flanged valves or other flanged connections. And the new UNITRACE Trace Cap permits quick, easy assembly of cross or tee connections.

### **Uses standard, preformed pipe insulation**

The efficiency of UNITRACE often makes insulation unnecessary. When it is needed, standard preformed insulation can be used. UNITRACE saves extra dollars by employing smaller diameter insulation than conventional systems.

### **Excellent corrosion resistance**

Thanks to the natural corrosion resistance of aluminum, UNITRACE is ideal for handling naval stores, molten sulfur, ammonium nitrate solutions, glacial acetic acid, fatty acids, tar, pitch, wax, urea, and similar products requiring heated transfer lines.

ALCOA UNITRACE and matching fittings are available in four standard pipe OD's: 1½", 2", 3" and 4". Get complete details on how you can use them to raise efficiency and lower cost on your own heated transfer lines. Call your nearest Alcoa sales office or write Aluminum Company of America, 892-M Alcoa Building, Pittsburgh 19, Pa.



### **Write for this FREE BOOKLET**

This fully illustrated booklet contains complete engineering, specification and fabrication data on ALCOA UNITRACE. Write for your free copy!



**ALCOA THEATRE**  
Fine Entertainment  
ALTERNATE MONDAY EVENINGS



## POSITIONS WANTED and AVAILABLE

- Active and Junior NACE members and companies seeking salaried employees may run without charge two consecutive advertisements annually under this heading, not over 35 words set in 8 point text type.
- Advertisements to other specifications will be charged for at \$10 a column inch.

### Positions Wanted

**Corrosion Engineer**—Twelve years' continuous experience in all phases of corrosion work including pipe lines, tanks, well casings, marine equipment etc. BS degree general engineering, registered professional engineer, married, age 34. CORROSION, Box 58-22.

### Positions Available

**Sales Engineer.** Opportunity of a lifetime. Progressive top rated Eastern Paint Manufacturer has splendid opening for experienced corrosion prevention salesman. Prefer young man with ability to manage, supervise and train salesmen in his department. Future possibilities unlimited and depending on his ability. Replies treated in strict confidence. CORROSION, Box 58-24.

**Mechanical or chemical engineer** position open for graduate engineer in sales work. Corrosion & chemical plant experience desirable. Reply CORROSION, Box 58-25.

## COATINGS SALES ENGINEER

Experienced, high performance coating sales and service engineer wanted for New Orleans Territory by major highly rated manufacturer of industrial coatings and linings. Must have history of successful sales. Degree in chemical engineering or chemistry preferred but not necessary. Base salary plus commissions. Maximum potential income high. Send complete information, details of experience, and recent photograph to O. A. Melvin, Carboline Company, P. O. Box 14284, Houston 21, Texas.

## CORROSION ENGINEERS

Several attractive openings are available with a highly regarded, major Eastern engineering company. These positions require Electrical Engineering graduates with 5-10 years experience in corrosion control investigations, corrosion testing and solution of corrosion problems on pipelines, underground cables, power plant structures, piers, wharves, water and oil storage tanks, etc.

These positions offer ample opportunity for professional growth and personal achievement. Considerable travel within the USA with possibilities of some overseas work. We invite you to investigate these opportunities by forwarding a complete confidential resume.

CORROSION, Box 58-17



Langlois



Barnwall



Glover

GREATER BOSTON Section Officers who were hosts to the Northeast Region Conference in October are shown. They are top left, Walter Langlois, section chairman also in charge of conference publicity; top right, Benjamin Barnwall, vice-chairman and conference registration chairman; and bottom, Manson Glover, secretary-treasurer and chairman of conference finance committee. Mr.

Glover helped compile a handbook on planning procedures for regional meetings.

## Houston NACE Member Honored by Kansas College

Edward T. O'Rourke, NACE member, was honored as outstanding alumnus of his 1938 class at St. Benedict's College, Atchison, Kansas. He was appointed Grand Marshall of the Homecoming Parade by the college president. He also presented the Homecoming Queen at half-time ceremonies during the October 10 football game with Pittsburg State College.

Mr. O'Rourke is affiliated with the Paint Division of Pittsburgh Plate and Glass Co., Houston.

## Cathodic Protection Talk

Installation of Deep Ground Beds for Cathodic Protection was the technical talk by W. F. Levert, United Gas Pipe Line, and Paul Miles, Interstate Oil Pipe Line, Shreveport, scheduled for the Shreveport Section, November 4.

## Section Discusses Coating

Applications and Limitations of Protective Coatings was the technical subject for the East Texas Section October meeting in Longview. The speaker was Tom O. Stephens, Amercoat Co., Houston.

## D E A T H S

Edward A. Suverkrop, one of the first members of NACE, died July 3. Personally known to many at Kure Beach and Wrightsville, N. C., testing centers, he directed corrosion studies the past 30 years for John A. Roebling's Sons Corp. of Trenton, N. J. He continued as consultant after retiring in 1956. He graduated from Columbia University School of Mines in 1913. He was associated with Chile Copper Company in South America and later with the Duriron Company.



## NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1959

Jan. 12-14—Canadian Region. Sheraton Mount Royal Hotel, Montreal, Quebec.

Feb. 11-13—Canadian Region. Calgary, Alberta.

March 16-20—15th Annual Conference and 1959 Corrosion Show. Sherman Hotel, Chicago.

Sept. 29-30, Oct. 1—Western Region Conference. Bakersfield Inn, Bakersfield, Cal.

Oct. 1-2—Southwest Region. Jacksonville, Florida.

Oct. 5-7—Northeast Region. Lord Baltimore Hotel, Baltimore, Md.

Oct. 12-15—South Central Region Meeting, Denver, Col.

Oct. 20-22—North Central Region, Cleveland.

1960

March—16th Annual Conference and 1960 Corrosion Show. Dallas, Texas, Memorial Auditorium.

Oct. 11-14—Northeast Region Meeting. Huntington, W. Va.

Oct. 25-28—South Central Region Conference, Mayo Hotel, Tulsa.

1961

March—17th Annual Conference and 1961 Corrosion Show. Buffalo, N. Y., Hotel Statler.

Oct. 24-27—South Central Region Conference, Houston, Shamrock Hotel.

1962

March—18th Annual Conference and 1962 Corrosion Show. Kansas City, Municipal Auditorium.

October 16-19—South Central Region Conference, San Antonio, Texas.

## SHORT COURSES

1959

January 22-23, Houston Section. Short Course on Fundamentals of Corrosion Control. University of Houston, Oberholtzer Hall.

February 11-13—Tulsa Section. Tenth Annual Pipe Line Short Course. Mayo Hotel.

March 31-April 12, Corrosion Control Short Course. University of Oklahoma-Central Oklahoma Section. North Campus, University of Oklahoma, Norman.

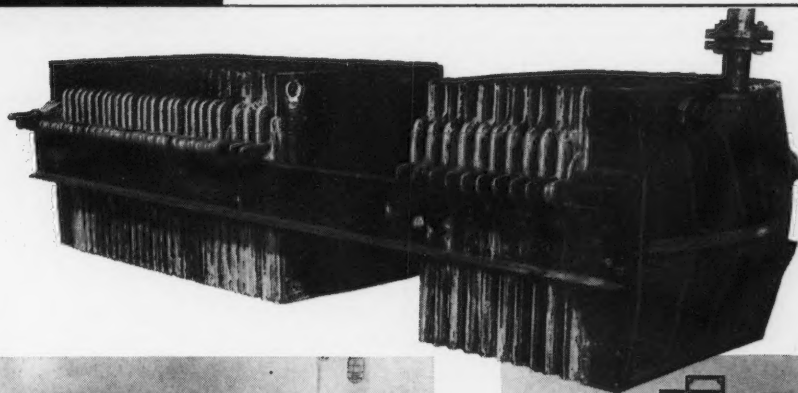
June—Teeche Section. Corrosion Control Short Course.

NACE regional and sectional secretaries who do not have a supply of the "News Report for Corrosion" forms may obtain copies from the Central Office. The form helps in providing a complete news story concerning meetings. The news deadline for CORROSION is the tenth of the month preceding month of publication.

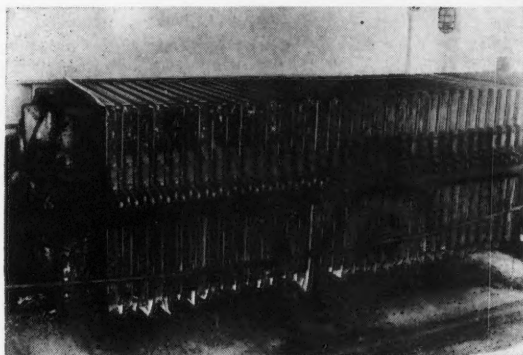


## CORROSION-RESISTANT WITH LAMINAC®

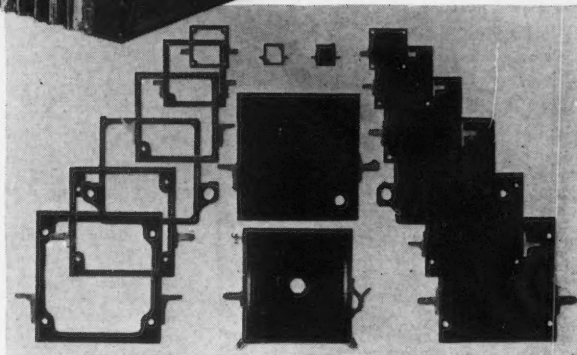
POLYESTER RESIN



THINNER, STRONGER LAMINAC plastic plates and frames provide greater filtering area and cake capacity than wood in filter presses.



LAMINAC HOLDS CLOSE TOLERANCES when ground, makes a perfect watertight seal when plates and frames are compressed between filter press heads.



ALL SIZES AND TYPES OF REINFORCED LAMINAC plates, frames and recessed plates are made by William R. Perrin Limited, Toronto, Canada.

*Reinforced LAMINAC  
filter plates and frames  
raise capacity up to 70%,  
cost less, last longer!*

For resistance to chemical attack, erosion and temperatures up to 240° F, nothing matches reinforced LAMINAC plastic filter plates and frames! Some of the plates illustrated, made by William R. Perrin Limited, Toronto, Canada, have been in service since 1953. They are still going strong in uranium, copper, zinc, and gold refining operations and in filtration of various fatty acids, pigment, dye, soap, pharmaceutical, vinegar, brine and pectin solutions.

Reinforced LAMINAC is far stronger than wood, making possible thinner plates with more filter chambers. These factors increase area and capacity as much as 70%. And while initially higher in cost than wood, LAMINAC lasts many times longer for lower net cost.

One-fourth the weight of metal, reinforced LAMINAC plates and frames are easier to handle. They cost much less. There is no coating to chip off. Product contamination is avoided, and the hard, smooth surface is easily cleaned.

For exceptional corrosion resistance and high strength in your tough applications, see your Cyanamid representative about LAMINAC!

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Los Angeles • Minneapolis • New York • Oakland • Philadelphia • St. Louis • Seattle  
In Canada: Cyanamid of Canada Limited, Montreal and Toronto



## TECHNICAL REPORTS

on

### HIGH PURITY WATER CORROSION

- Symposium on Corrosion by High Purity Water by Committee T-3F on High Purity Water which includes:  
Introduction to Symposium on Corrosion by High-Purity Water by John F. Eckel.  
Corrosion of Structural Materials in High-Purity Water by A. H. Roebuck, C. R. Breder and S. Greenberg.  
Corrosion Engineering Problems in High-Purity Water by D. J. DePaul.  
The Importance of High-Purity Water Data to Industrial Applications by W. Z. Friend. Per Copy.....\$1.50
- Symposium on Corrosion by High Purity Water. Five Contributions to the Work of NACE Technical Committee T-3F on High Purity Water. Pub. 57-22.  
Measurement of Corrosion Products in High Temperature, High Pressure Water Systems by A. S. Sugalski and S. I. Williams.  
Corrosion of Aluminum-Nickel Type Alloys in High Temperature Aqueous Service by F. H. Krenz.  
Corrosion of Aluminum in High Purity Water by R. J. Lobsinger and J. M. Atwood.  
The Storage of High Purity Water by Richard R. Dlesk.  
Water Conditions for High Pressure Boilers by D. E. Voyles and E. C. Fiss. Per Copy.....\$1.50

Remittances must accompany all orders for literature the aggregate cost of which is less than \$5. Orders of value greater than \$5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65¢ per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

## NATIONAL ASSOCIATION OF CORROSION ENGINEERS

1061 M & M Bldg. Houston 2, Texas

## Central Oklahoma Appoints Short Course Committee

Men who will plan and produce the corrosion control short course to be presented March 31-April 2 at Oklahoma University by the Central Oklahoma Section and the Extension Division of the University of Oklahoma have been named to the short course committee.

Committee members are Dan. H. Carpenter, Atlas Powder Co.; Frank B. Burns, General Asphalts, Inc.; H. B. Carlile, Jr., Shell Oil Co.; John Daly, Sunray-Midcontinent Oil Co.; M. L. Steffens, Oklahoma Natural Gas Co.; Ralph J. Rice, Magnolia Petroleum Co.; Ted V. Sherman, Oklahoma University Extension Division; and R. V. James, Oklahoma University engineering department.

The committee plans to meet during the afternoon prior to each Central Oklahoma Section meeting.

## Dates Through May Set By Cleveland Section

Technical subjects and dates for meetings through May have been set by the Cleveland Section. Papers and talks will emphasize coatings since Cleveland is a paint manufacturing center.

Meeting dates and subjects announced are as follows:

December 16—Stainless Steel, by W. Luce, Duriron Co.

January 20—joint meeting with the Electrochemical Society.

February 17—Linings, by a speaker from Heil Process Equipment Co.

April 21—Protective Coatings, by Stanley Lopata, Carboline Co.

May 19—Panel Discussion, R. West as moderator.

The Cleveland Engineering and Scientific Center has been selected as the meeting place.

William Hooker, Harco Corp., was added to the section's advisory committee to replace J. A. Pantalone, who has moved to California.

Protective Coatings, by George Richardson, Glidden Co., was scheduled for the November meeting; Coatings Specifications, Application and Performance, by George F. Orr, Hill-Hubbell & Co., for the October meeting.

Section members made a trip through the laboratories of the National Advisory Committee for Aeronautics for the September meeting.

## New Officers to Be Elected By San Francisco Section

New officers for the San Francisco Bay Area Section will be elected this month by letter ballot. Candidates nominated were John P. Fraser, Shell Development Co., chairman; Roy Dean, Pacific Gas and Electric Co., vice-chairman; and John Caldwell, Bunker Hill Co., secretary-treasurer.

Provision was made for nominations to be made on the ballots by the members.

The Use of the Corrosometer for Refinery Corrosion Control was the technical talk scheduled for the December 10 meeting. Speaker is to be Robert L. Piehl, Standard Oil Company of California.

Comparative Properties of Five Metal Protective Coatings, by Roy Landis, Sherwin-Williams Co., was the November meeting's subject.

## Cathodic Protection Panel

A panel discussion on cathodic protection was the November technical program for the Lehigh Valley Section.

At the September meeting, 15 members and 13 guests heard Spencer W. Shepard, Chemical Construction Corp., speak on Corrosion in the Chemical Industry.

## Paint-Varnish Club Meets

A paper on Monastral red pigments was presented by Thomas B. Reeve, Pigments Department, E. I. du Pont de Nemours & Co., Inc., at the October 13 meeting of the Houston Paint and Varnish Production Club.

The University of Oklahoma Corrosion Control Short Course will be held March 31-April 12 at the North Campus, Norman.

## Deeds

"Tis deeds must win the prize"

—Shakespeare

The prize in virtually every business operation is continuing acceptance of a product or service.

And this prize is achieved in only one way—by reliable performance.

For 29 years, Mayes Bros. has performed deeds of dependability for its customers. For 29 years, these customers have been calling on Mayes Bros. repeatedly for pipe coating and wrapping jobs that will stand up under the rigors of underground service.

Mayes Bros. values this "prize" above all others and will continue to put permanence in pipe on every job with materials and workmanship of consistently high quality.

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## COKE BREEZE

### Backfill for Anodes

Ideally suited for use with anodes. Has a high carbon content and comes in sizes of 1/8-inch x 0 to 3/8 x 1/2 inch. In bulk or sacks. Prices on other sizes on request.

### National Carbon Anodes

### Magnesium Anodes

### Good-All Rectifiers

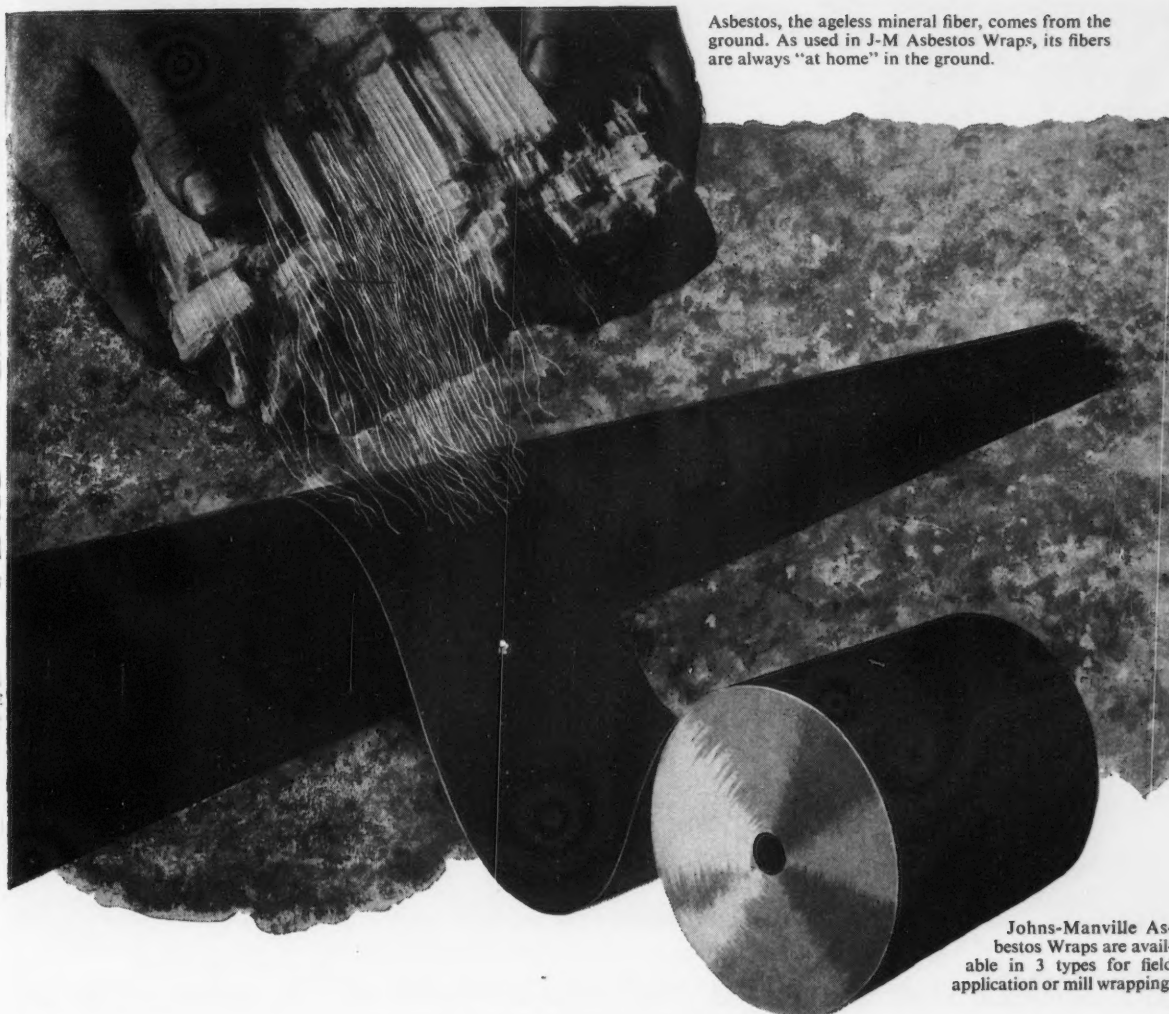
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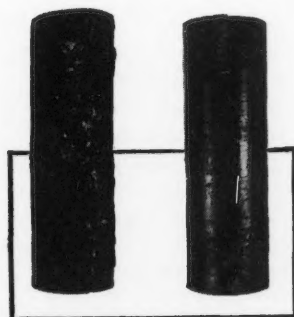


Asbestos, the ageless mineral fiber, comes from the ground. As used in J-M Asbestos Wraps, its fibers are always "at home" in the ground.

Johns-Manville Asbestos Wraps are available in 3 types for field application or mill wrapping.

LIKE WRAPPING YOUR PIPE ENAMELS IN STONE!

## ... J-M Asbestos Wraps for tough, lasting protection



(Left) Plasticized enamel without J-M Asbestos Wrap did not stand up in soil stress tests. Coating had been applied to 18" sections of 4" O.D. pipe buried in bentonite clay which was then subjected to a series of wetting-downs and dryings.

(Right) Plasticized enamel with J-M Asbestos Wrap shows the full effectiveness of shielding action provided. Sample was in excellent condition after 28 wetting-drying cycles.

Obviously, pure stone can't be fabricated into sheet or roll form for pipeline enamel protection. But you can get the closest thing to it—J-M Asbestos Wrap, made of a lasting mineral fiber that's completely at home underground and uniquely conditioned by nature not to rot or decay.

In addition to being ageless, asbestos fibers are unusually strong and tough. As used in J-M Wraps, the fibers are felted, then impregnated with either coal tar or asphalt saturant to form, virtually, a flexible covering of stone. These "stone-like" qualities provide the durable shielding needed to

protect enamels from earth loads, soil movement, other forces which weaken coatings and permit pipe corrosion to set in.

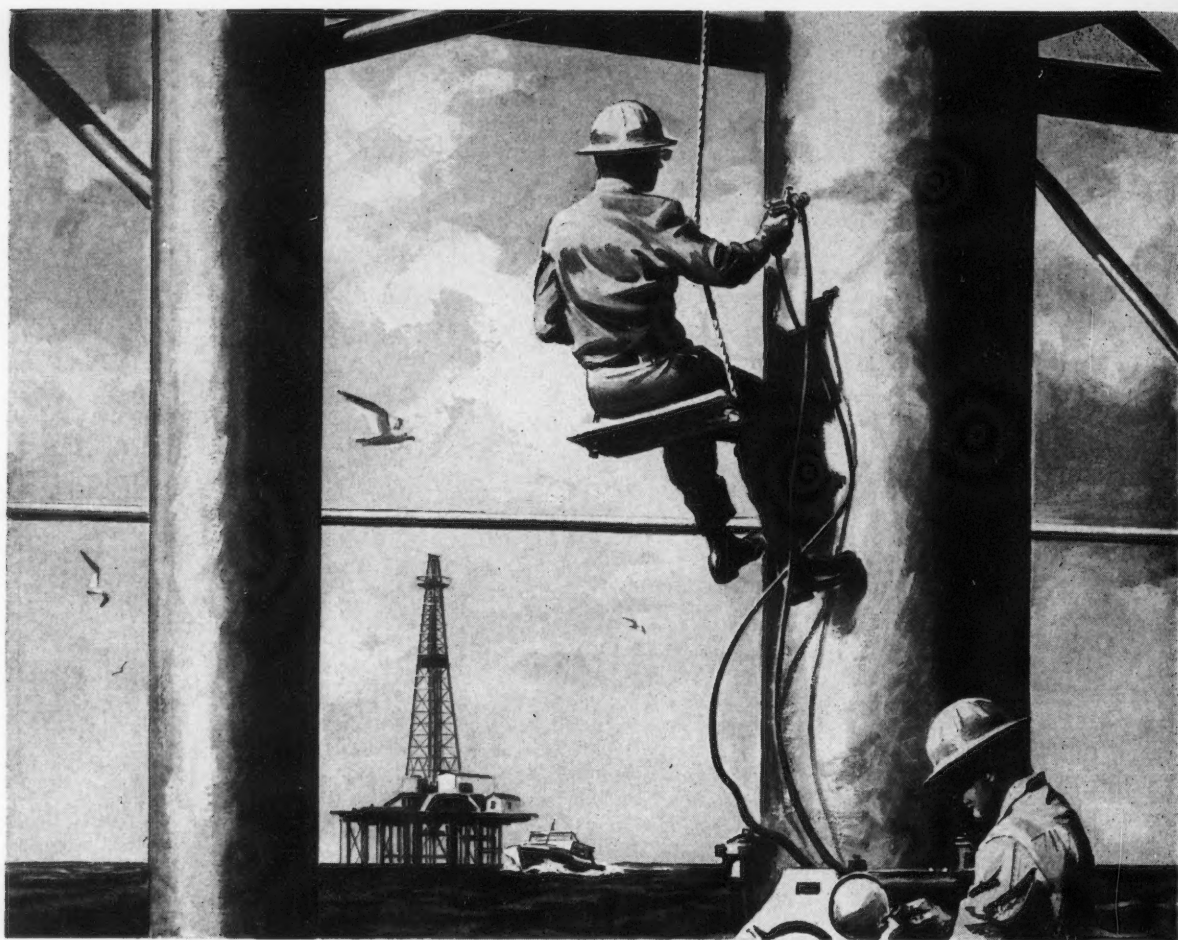
Extensive research and field tests conducted by a leading firm of consulting engineers have demonstrated these long-lasting protective values of J-M Wraps: "Asbestos Wraps offer the most effective single protection against damage—prolong the working life of pipeline enamels."

Pipeline engineers are invited to write for their own copy of this report PP-34A, a 3-Point Study of Pipeline Coatings and Wrappers, at no cost or obligation.

Johns-Manville, Box 14, New York 16, N. Y. In Canada, Port Credit, Ontario

# JOHNS-MANVILLE





## **OFFSHORE** the durable corrosion-resisting qualities of Humble's coatings give substantial savings!

On costly offshore drilling platforms and equipment, Humble's protective coatings have proved their ability to resist the powerful corrosive action of salt water splash, spray and vapor. There's a Humble protective coating for every surface from the splash-zone to the crown block, including the working platform and structures on the platform. Humble's complete line of protective coatings includes RUST-BAN®, vinyls, epoxies, phenolics, alkyds and others, available in a wide range of finish colors.

For the platform itself, excellent results have been obtained by using a six-coat vinyl system, including one vinyl mastic coat, to a total dry film thickness of from 14 to 16 mils. Humble's vinyl protective coatings produce a tough, flexible film that gives remarkable resistance to corrosion and weathering.

Humble protective coatings are readily available at Humble wholesale plants throughout Texas and New Mexico.

• For detailed information on how Humble's protective coatings can save you money, call your Humble salesman, or phone or write:

Humble Oil & Refining Company  
Sales Technical Service  
P. O. Box 2180  
Houston 1, Texas

HUMBLE OIL & REFINING COMPANY

**HUMBLE**





## GENERAL NEWS

### Armour Foundation Corrosion Research Surveyed in Report

The following corrosion research programs are reported in the 1957 annual report of the Armour Research Foundation:

#### Coatings

The number of ceramic materials used as coatings is being increased. Process equipment has been developed, and such coating compositions as stabilized zirconia, rutile titania, alumina, ilmenite and mullite have been standardized.

National Carbon Company is studying protective treatments and coatings for graphite. Practical application of graphite as a refractory is limited by its susceptibility to oxidation at temperatures above a red heat.

#### Air Pollution

An air pollution evaluation survey has been conducted at Gary, Ind. Analysis of the data will aid development of a modernized program of air pollution control.

#### Inhibitors

Research at the Wright Air Development Center's Organic Materials Branch is concerned with the synthesis of organic chemicals and their evaluation as high-temperature oxidation-corrosion inhibitors for mineral oil, synthetic ester, silane and silicone-type fluids.

#### Metallurgy

Boeing Airplane Company and the Air Force are determining the tension, compression, bearing and shear properties of 6Al-4V titanium alloy at temperatures from 75 to 1000 F. Results will provide information to be used in future aircraft and missile structures using the alloy.

Evaluation of the effects of time, temperature and stress on the stability of titanium-chromium alloys has been completed by Wright Air Development Center.

A study of Kanigen R chemically-deposited nickel plating has been undertaken by General American Transportation Corporation.

#### Electrochemistry

Investigation of basic factors affecting tinplate corrosion is being conducted by Jones and Laughlin Steel Corporation.

Development of electrochemical techniques to improve the corrosion resistance of tinplate containers is being studied at Continental Can Company. New laboratory tests for evaluating and comparing enamels have reduced the time of long-term pack tests.

Procedures to inhibit white rust formation on hot-dip galvanized steel are being developed by Weirton Steel Company, division of National Steel Corp.

Vacuum deposited ductile films on mild steel surfaces are being studied by

U. S. Steel Corporation. Test panels have satisfactorily withstood peeling, bend and rusting tests.

#### Welding and Brazing

Effects of interstitial contaminants, singly and in combination, on the weldability of titanium alloys have been studied at the Wright Air Development Center. Effects of carbon, oxygen, nitrogen and hydrogen have been investigated.

The Center has also started a brazing filler metal alloy development program concerned with the joining of stainless steel to Inconel.

### Wrought Iron Standards Set by Fire Underwriters

Standards for the use of wrought iron for filling, inside and mill-use piping in the construction and installation of water tanks have been issued by the National Board of Fire Underwriters.

Wrought iron was designated in the standards because of its resistance to corrosion caused by wet and dry condition, foreign elements in water and general atmospheric conditions. The inaccessibility of tank piping requires a durable material to keep maintenance at a minimum.

These standards were also recommended by the National Fire Protection Association.

### New 16mm Color Film On Corrosion Available

A new 16mm color film on corrosion explains what causes anodes and cathodes to form on steel surfaces, how they produce electrolytic corrosion and what can be done to prevent this corrosion.

Available from Amercoat Corp., 4809 Firestone Blvd., South Gate, Cal., this sound-track film discusses inhibitors, alloys, galvanizing, cathodic protection and coatings. Case histories are presented to show how corrosion problems are solved in various industries.

Running time is 19 minutes.

## BOOK NEWS

**Guide to Technical Reports.** By Henry Dan Piper and Frank E. Davie. 82 pages, 6 x 9 inches, paper. May 21, 1958. Rinehart & Co., Inc., 232 Madison Ave., New York 16, N.Y. Per copy, \$1.

A well-organized brief guide to the preparation and writing of reports on technical subjects. The subject matter includes fundamentals of a report, writing methods, types, standard procedures, punctuation, grammar and style, and an appendix of examples of the several kinds of reports and their revisions.

The guide is a revision of an earlier book prepared by the authors for use in Shell Oil Company and in classes for students at California Institute of Technology.

### Longest Offshore Platform To Have Cathodic Protection

A Y-shaped offshore platform one mile in length will have its steel pilings protected from salt water corrosion by rectifier-type cathodic protection.

Believed to be the world's longest, this offshore platform is being built at the Freeport Sulphur Company's Grand Isle Mine off the coast of Louisiana near Grand Isle.

The New Orleans division of Corrosion Rectifying Company is in charge of cathodic protection of the platform. Coreco-type rectifiers with graphite anodes are being used. The project will probably require over a year for completion.

Sections of the platform will be pre-assembled in port and towed to the mine site for final assembly.

### Co-op Educational Programs Discussed in New Book

Co-operative scientific educational programs, under which students alternate work with study in their chosen fields, are discussed in a new book entitled "Hand in Hand," published by Gordon & Co., Bedford 56, Mass.

Actual work in the student's chosen field in industry is alternated with periods of college study to balance theory with experience and provide a broader base for success. This program is one answer to modern education's problem of how to steer students into scientific careers for which they are best suited, according to the committee of engineers and educators which authored this book.

### Coating Analysis Published

Time-saving techniques for infrared analysis of coatings are described in an Army research report just released to industry.

Entitled "Rapid Determination of Polyacrylonitrile in Coatings by Infrared Absorption Spectrometry," the report is available from the Office of Technical Services, U. S. Department of Commerce, Washington 25, D.C., at 50 cents per copy.

### Corrosion Resistance Tables

A new reference catalog on stainless steel pipe fittings includes a technical section giving corrosion resistance tables. Data on manufacturing standards and welding of stainless steel are also included. The catalog is available from Ladish Co., Cudahy, Wis.

### Chemical Industry Society

Meetings of the Corrosion Group of the Society of Chemical Industry in London, England, have been scheduled for Dec. 19, Jan. 6, 16 and 22, Feb. 11, March 4 and 18, April 8, and May 6.

NACE members visiting in England have been invited to attend the meetings.



## Equipment Services

# NEW PRODUCTS

## Materials Literature

### Adhesives

**High-strength Adhesives** that bond a variety of material to each other or to dissimilar materials have been developed by Ceilcote Company, Cleveland. Called Ceilcote Adhesive No. 348, it is recommended for bonding new concrete to old concrete and can be used to bond steel, brass, aluminum, wood and certain plastics to each other or to concrete.

### Aluminum

**World's First** all-aluminum railroad refrigerator cars are being tested by the Canadian National Railways. Designed by Aluminum Company of Canada, Ltd., and National Steel Car, the new cars were designed with all-aluminum construction to eliminate expensive repairs and repainting due to corrosion caused by brine solutions used in the refrigeration system. The use of aluminum reduced the gross weight of the car seven tons over standard rail cars.

### Cleaning

**Cleaning Costs** for metals can be analyzed and compared by a new method presented in a booklet issued by the Du Pont Electrochemicals Department, Wilmington, Del. Total hourly cleaning costs can be figured by inserting basic operating information into simple formulae.

**Chelating Agents** are used in a new technique for hydroxy apatite boiler scale removal announced by Dow Chemical Co., Midland, Mich. Downtime has been reduced as much as 30 hours for high make-up industrial boiler cleaning. The agents are introduced into the boiler system as a water solution and circulated at low pressures until the reaction is complete.

**Ultrasonic Cleaning Systems** in larger, high-powered sizes are being mass produced by Narda Ultrasonics Corp., Westbury, Long Island, N. Y. Known as the Series 5000 SonBlaster, the systems include an output generator and a 10-gallon transducerized cleaning tank.

### Coatings

**Wire and Powder Sprayed Coatings** are discussed in a new engineering data bulletin published by Metallizing Engineering Co., Inc., Westbury, Long Island, N. Y. Characteristics and applications of the coatings are given plus various types of spray equipment.

**Galvanized Surfaces** can be protected by a new paint with a red lead-iron oxide-zinc dust pigmentation in an alkyd resin vehicle. The paint can be used on rusted, weathered, treated or new galvanized surfaces. It is discussed in Red Lead Technical Letter No. 13 available from Lead Industries Association, 60 East 42nd Street, N. Y. 17.

**Clear and Colored Coatings** for use on metal, plastics, glass and wood are ex-

plained in a brochure published by Bee Chemical Company, 12933 South Stony Island Ave., Chicago 33. Information on coatings for various surfaces by spraying, screening and roller coating is given.

**Chrom-Lume Process**, a new method for plating chromium directly to aluminum and its alloys, has been developed by Service Hard-Chromium Co., Route 22, Union, N. J. It provides direct coating of chromium to the basic metal with no intermediate metallic deposition of copper, nickel or zincate needed.

**Kanigen Process** is a method for depositing a uniform corrosion and abrasion resistant nickel alloy coating on iron, copper, aluminum, titanium and their alloys without the use of electricity. Details of this process are given in Technical Bulletin 258 available from General American Transportation Corp., 135 South La Salle, Chicago 90.

### Drum Vent

**Automatic Pressure Relief** for 55-gallon steel drums is provided by a drum vent valve manufactured by Protectoseal Co., 1920 South Western Ave., Chicago 8. Designed for drums with 2-inch bung openings in the head, this valve prevents drum rupture and loss of contents even in extreme conditions when the drum is surrounded by fire.

### Fittings

**Stop-N-Go** is a new fitting that serves as a line cap and as an extension fitting for 2-inch gas mains. Developed by Dresser Mfg. division, Dresser Industries, Inc., Bradford, Pa., the fitting permits the making of extensions without shutting off the main if pressures do not exceed 30 psi.

### Instruments

**Technicon AutoAnalyzer**, a new system for continuous automatic chemical analysis, can detect trace materials down to parts per billion with an accuracy of 1 percent, according to Technicon Controls, Inc., Chauncey, N. Y. All steps of chemical analysis (measuring, mixing, purifying, processing, comparing and recording) are done by automation.

### Laboratories

**Planning and Development** of new products are emphasized in the newly created department at Metal & Thermit Corp., 100 Park Ave., New York 17. Present products of the company include electroplating, organic coatings, welding supplies and products for the ceramic industry.

**Industrial Technical Service**, a new customer service division, has been added to Octagon Process, Inc., Staten Island 1, N. Y., manufacturers of metal clean-

ers, paint strippers, rust and carbon removers and pickling inhibitors.

### Metals—Ferrous

**Zinc-coated Sheets** of high strength, low alloy steel are being produced by Jones & Laughlin Steel Corp., Pittsburgh 30. With a yield point of 50,000 minimum psi and tensile strength of 70,000 minimum psi, the product will be marketed as High Tensile Low Alloy JALZinc Sheets. Principal uses will be for structural applications such as roofing, siding, flooring, angles and channels. Its corrosion resistant qualities make it suitable for use as highway guard rails.

**Proper Specifications** can be determined for various types of carbon, alloy and stainless steel tubing, pipe, fittings and flanges from a folder published by Babcock & Wilcox Co., Tubular Products Division, Beaver Falls, Pa. The folder serves as a cross reference between the type of steel, the application and the specifications of the product.

**Wrought Iron** is being used in construction of the Thomas H. Allen Electric Generating station on the shore of Lake McKellar, south of Memphis. Specified as one of the materials in this large circulating water system, wrought iron is used for piping and elbows leading into the condenser and concrete manifold on the discharge side.

### Pipe

**Plastic Irrigation Pipe** for underground use is being manufactured by Southwestern Plastic Pipe Co., P. O. Box 117, Mineral Wells, Texas. An engineering data bulletin is available giving velocity and head-loss data for usage ranges of 200 to 5200 gallons per minute.

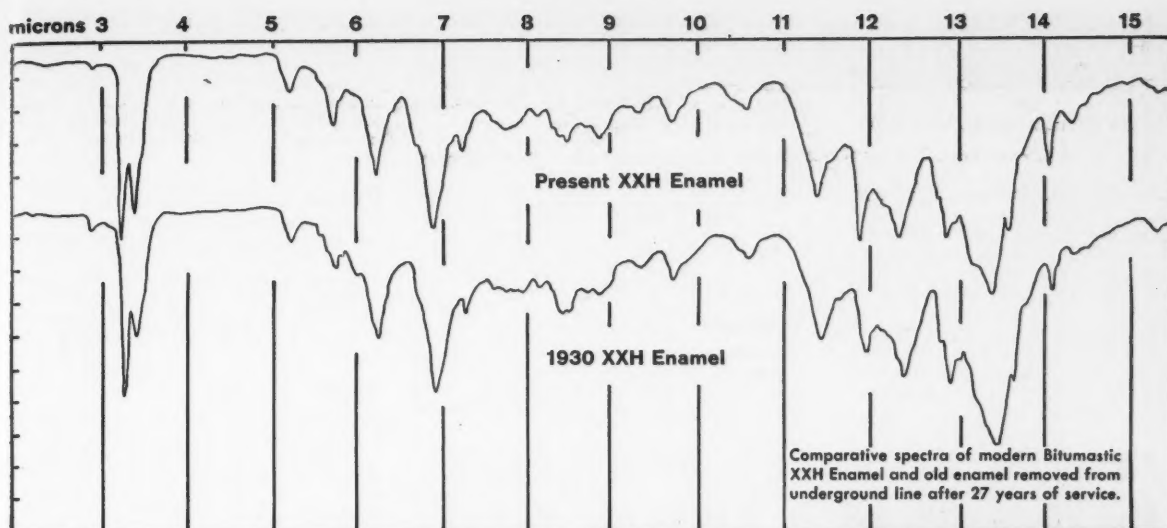
**Carlton PVC Pipe and Fittings**, a 16-page booklet describing the pipe, tubing and fittings made by Carlton Products Corp., 10225 Meech Ave., Cleveland 5, Ohio is available on request. Tabulated data are given on resistance to corrosives.

**Impervite Piping System**, using glass fiber armor for protection against physical abuse, can be fitted easily in the field with only a hacksaw and a wrench because it is made of impervious graphite. Available from Falls Industries, Inc., Aurora Road, Solon, Ohio, the pipe and fittings are furnished with flanges attached. Joining is accomplished by gasketing between flanges.

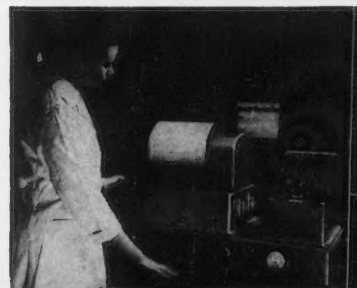
### Plastics

**Silicon Rubber Parts** are being precision molded by Sierra Engineering Co., Sierra Madre, Cal. Parts are formulated for extreme conditions including resistance to fuels, oils, solvents, moisture, ozone and weathering.

**Weyman Company**, a northern California representative for Plasite, a product (Continued on Page 114)



## Here's dramatic proof of modern coal-tar enamel's long-lasting corrosion protection



Natural Gas Pipeline Company of America recently relocated a portion of its line near Truro, Iowa which had been in continuous service for 27 years. Inspection of the pipe disclosed that it had been completely protected against corrosion by its Bitumastic XXH Enamel protective coating. In fact, the pipe—under minimum cathodic protection for only 2 of its 27-year life—was in such excellent condition that the firm planned to re-use this entire portion of the line.

A section of this old pipeline and its coating was brought to the Verona Research Laboratories of Koppers for testing and analysis. The 27-year-old enamel was subjected to standard ASTM tests for melting point, penetration, ash content and other physical characteristics. All the test results showed that the enamel had not been affected by its underground service and that it is identical to material being supplied today.

Finally, the infra-red absorption spectra shown above were obtained: note the close parallel between the old and new Bitumastic enamels throughout the spectrum. This similarity indicates that present-day Bitumastic enamel will give the same protection against corrosion underground as did the old enamel. Modern coal-tar enamel can be expected to *exceed* the 27-year record of the old coating, since today's application and inspection techniques are far better than those used in the '30s.

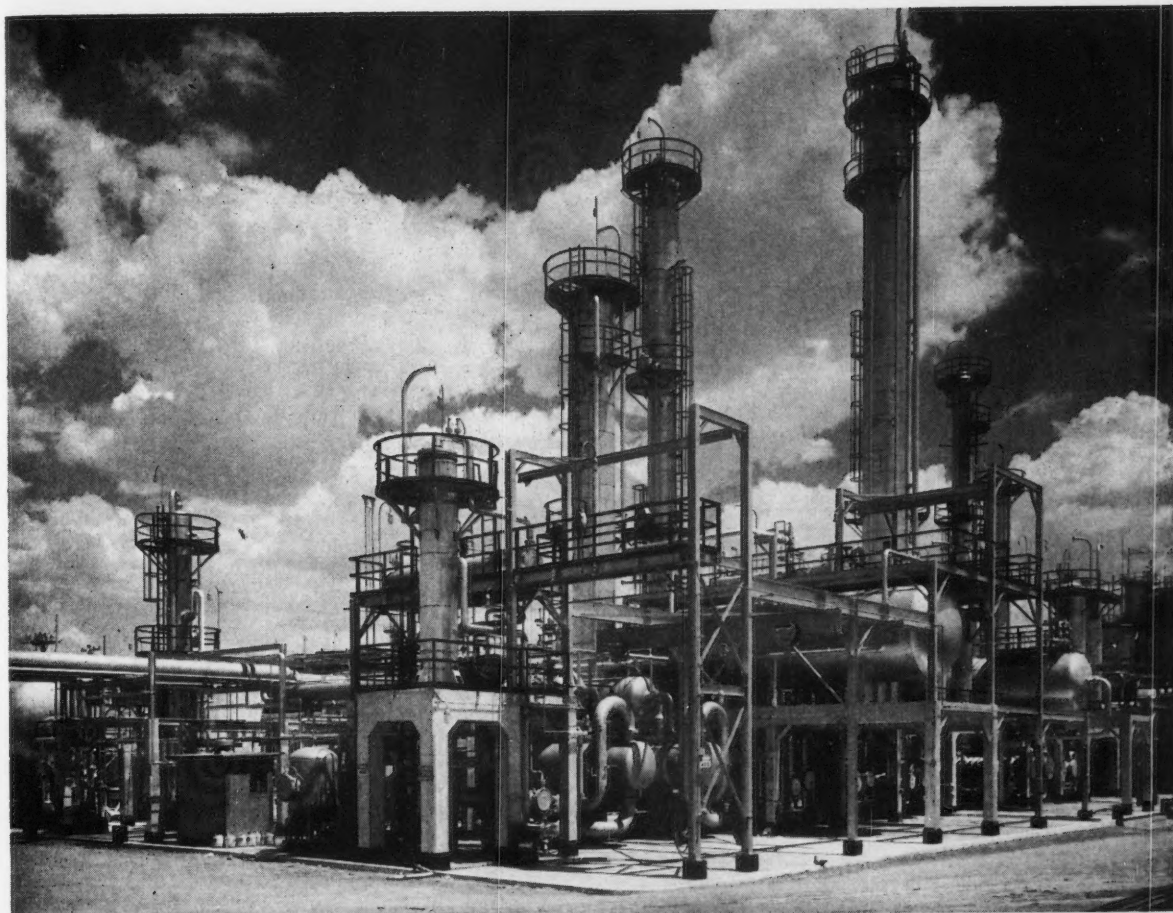
This proof-of-protection confirms again the superior waterproofing ability of coal-tar enamel that makes it the best investment for permanent protection of your pipeline. For further information, write or call Koppers Company, Inc., Tar Products Division, Pittsburgh 19, Pa. District Offices: Chicago, Los Angeles, New York, Boston, and Woodward (Birmingham), Ala. In Canada: Koppers Products Ltd., Toronto.



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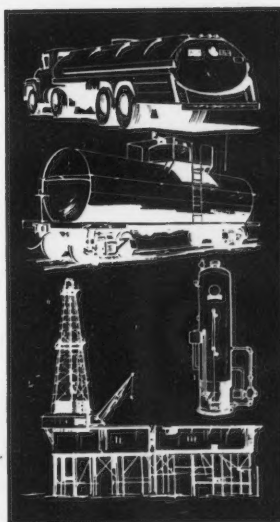


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## NEW PRODUCTS

(Continued From Page 114)

dimensions, Rovalves are manufactured by W. G. Rovang & Associates, Inc., 1945 N. Columbia Blvd., Portland 17.

**Positive Choke Flow Control** employing a patented access fitting principle is being produced by Cosasco Division of Perfect Circle Corp., El Monte, Cal. This control eliminates expensive valves from oil production flow lines and contains no moving parts subjected to pressure, fluid, distortion or wear. Choke beans can be changed under full operating pressures without system shutdown.

**Crosby Style JB Valve**, a newly designed relief valve manufactured by Crosby Steam Gage and Valve Co., Wrentham, Mass., has special bellows to seal out corrosion and to prohibit foreign particles building up and freezing the moving parts. Made of corrosion resistant alloys, the bellows permit valve springs and guides to maintain correct size even in corrosive or fouling services.

**Tiny**—a new solenoid valve only 3½-inch in height—has been introduced by Atkomatic Valve Co., Inc., 545 W. Abbott Street, Indianapolis 25. Designated Type TT, it is a single-seated, angle-type valve machined from brass bar stock with stainless steel plunger and point assembly. Direct lift operation makes it suitable for operations under wide ranges of pressures.

**Chem-Check Valves** are all plastic, available in PVC types I and II, Kralastic, Penton and Profax. They have such mechanical features as full-flow, low pressure drop and built-in vibration damper. The valves can be operated in any position and are available in pipe sizes from ½ to 2 inches from Chemtrol Company, Lynwood, Cal., manufacturers of plastic piping equipment for corrosion services.

**Flex-Plug Plastic Gate Valves** with socket-weld ends have been announced by Vanton Pump & Equipment Corp., Hillside, N. J. Available in sizes from ½ to 2 inches in both PVC or styrene-copolymer, these valves offer the combined features of straight through no-pressure-drop flow with close throttling control.

## Welding

**Thermoweld** electrical connectors to make connections of cathodic protection wiring are described in a pamphlet available from Continental Industries, Inc., 7401 East 41st St., Tulsa, Okla. The system involves the use of powdered copper oxide and aluminum which produce a fusion weld inside a mold.

**Recommendations for Arc-Welding Titanium** gives comprehensive information to persons interested in the techniques. The brochure may be obtained from Mallory-Sharon Metals Corp., Niles, Ohio.

## Water Treatment

**Water Service Laboratories, Inc.**, 615 West 131st Street, New York 27, will treat the water to prevent corrosion in the air conditioning system of the new 34-story gold-colored skyscraper being built at 575 Lexington Avenue. The cooling system will have a capacity of 2000 tons.

## MEN in the NEWS

**Ralph Rathyen** has been promoted to industrial sales manager of Joseph Dixon Crucible Co., Jersey City, N. J.

**Wesley D. Schroeder** has been promoted to product research manager in the Research and Development Department of Pittsburgh Coke & Chemical Co.

**Robert B. McQuarrie** has been assigned as northwest coast field service engineer for A. M. Byers Company.

**Joseph F. Santilli, Jr.**, has been appointed marketing manager of Atlas Powder Company's Aquaness Department in Houston.

**Calvin L. Wright** is field service engineer for A. M. Byers Company in Baltimore. He succeeds V. H. Ross who retired after 28 years' service.

**Edward B. Saubestre** has joined Enthone, Inc., New Haven, Conn., as assistant research director. His work has been in metal finishing and electrochemistry.

**Eldridge K. Camp**, manager of the Finishes and Corrosion Laboratory at IBM, participated in the December 2-4

(Continued on Page 118)

**FIND**  
the HOLIDAY  
the SHORT  
the PIPE

with **TINKER & RASOR'S** New  
**PEARSON-TYPE**  
**Model PD, Detector**

Tinker & Rasor have developed a new, compact, highly effective, all transistor, Pearson-type detector. This instrument, the Model PD, produces a 15 watt, 750 cycle audio signal on a buried pipeline. This signal can be clearly heard through a new T & R 4-stage, transistor receiver, small enough to be worn on the operator's belt. Through sharp filters, this new

receiver rejects unwanted 60 cycle signals. The receiver weighs but 14 oz. and draws 800 micro amperes from a 4-volt mercury battery. The Model PD detector makes it possible to follow a buried pipe, to locate holidays in that pipe's protective coating as well as any electrical shorting between the pipe and other metal structures.

All components except oscillator storage battery are supplied in a sturdy carrying case.

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with Inter-  
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operator's belt.

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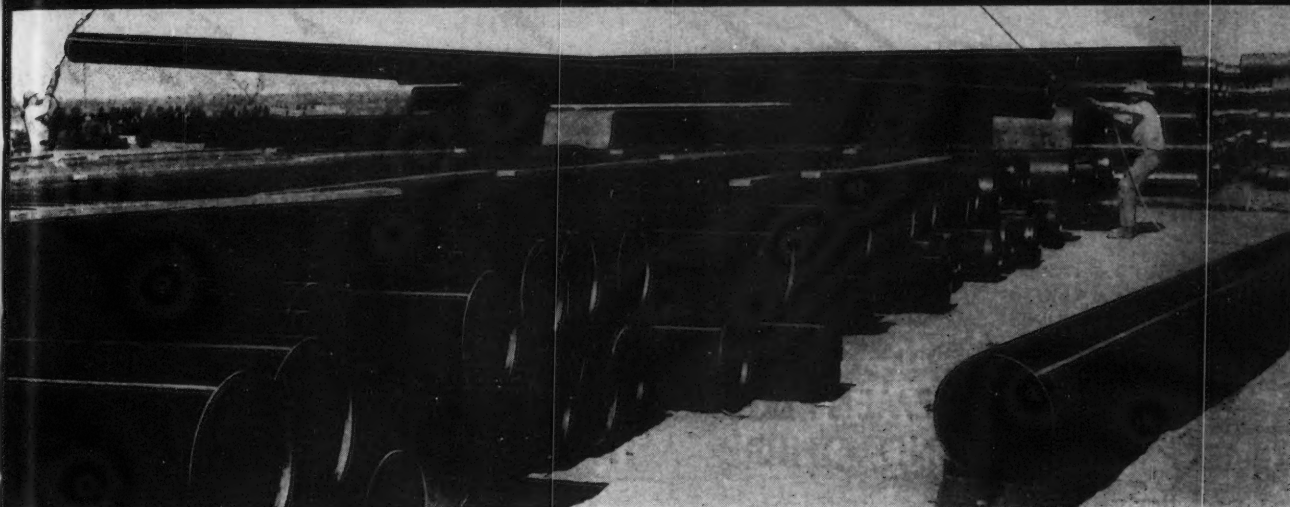
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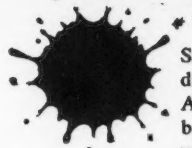
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*Now, lay coated pipe without "Special" handling*



ABOVE: 16-inch pipe being spray-coated for use in new Transcontinental Gas Pipe Line Corp. gas-gathering system near Tilden, Texas.



Save up to 20 per cent in laying costs on large-diameter pipelines. Spray-coat pipe with Copon Arocoat before installation. Then, haul it, stack it, bend it and lay it with little more care than you would use for bare pipe. Tough Copon Arocoat coatings will survive these operations without chipping or cracking, and Copon Arocoat can be applied to welds with an ordinary paint brush.

A combination of epoxy and coal tar resins, Copon

Arocoat coatings provide superior corrosion control. They will not deteriorate in the ground... remain tough and elastic... and are unaffected by temperature extremes. Successful applications include submerged, buried and exposed transmission lines.

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FOR CORROSION CONTROL



## MEN in the NEWS

(Continued From Page 116)  
conference on reliable electrical connections held in Dallas by the Electronic Industries Association. He is a member of NACE.

**James A. MacDonald**, a NACE member, has been named A. M. Byers Company representative in Toronto, Ontario, Canada.

**John A. Kittrell** has been named manager of American Viscose Corporation's Meadville, Pa. acetate plant. Mr. Kittrell, immediately afterward made the following appointments, **Harley J. Morris**, technical adviser to the plant manager; **William K. Mohny**, technical

superintendent; **Anderson C. Nalley, Jr.**, head of the processing department; and **Joseph M. Hoegerl**, chief control chemist.

**Robert W. Titgemeyer** has been named branch manager of the Los Angeles territory by the Carpenter Steel Co., Reading, Pa. He started with Carpenter as sales engineer in 1947.

**Owen H. Walker** has been appointed Houston and Gulf Coast district sales manager for the Ohio Injector Co. After graduating from the University of Houston in 1951 he became associated with Gulf Oil Co. in sales and supervisory assignments.

**Louis G. Glesmann**, who has been vice president and general manufacturing manager as well as a director of Revere

Copper and Brass Inc., was scheduled for retirement August 31 after 43 years of service. Mr. Glesmann, who is 65, will continue with Revere in a consulting capacity.

**Charles W. Sherman** has been appointed technical director of Jones & Laughlin Steel Corp., stainless and strip division. He will be in charge of the division's research and development activities.

**Edward L. Kent, Jr.**, has been named industrial sales manager of Kohler-McLister Paint Co., Denver. A NACE member, Mr. Kent will co-ordinate all industrial coating sales. Kohler-McLister is a member of Copon Associates, manufacturers of epoxy coatings for corrosion control.

**Frank I. Dorr, Jr.**, has joined the corrosion engineering staff of Rio Engineering Company, Houston. **Ben Russell** is a new member of the sales staff. Both men are NACE members and have several years experience in the corrosion engineering field.

**T. W. Farrer**, formerly of the Chemical Research Laboratories of DSIR, has joined Spencer and Partners, Consulting Engineers, 19 Grosvenor Place, London, S.W.1. Mr. Farrer has recently completed a corrosion research program at Cambridge University.

**Richard H. Shaffner** has been named sales manager of the northern division of Prufoat Laboratories, Inc., Cambridge, Mass. His area will include Buffalo, Pittsburgh, Cleveland, Cincinnati, Detroit, Chicago and St. Louis with headquarters in Niagara Falls, N. Y. A NACE member, Mr. Shaffner has been active in the Niagara Frontier Section.

**E. R. Camarena** will be the technical consultant for California Chemical International, Inc., in South America. His office will be in Sao Paulo, Brazil. **Ivan Breval** will replace Mr. Camarena on the CCI San Francisco sales staff.

**Robert N. Smith**, vice president of Tempco, Inc., Nashville, Tenn., was elected president of the Porcelain Enamel Institute at its annual meeting in September.

**Dr. Marvin O. Shrader**, supervisor of organic chemicals research for Pittsburgh Coke and Chemical Company, died September 21.

**Herman E. Bakken**, vice president of Aluminum Company of America and general manager of the refining division, has retired and will be replaced as manager by **J. Ranald Fox**.

**S. S. Isquith**, U. S. Navy rear admiral retired, has been appointed vice president of the board of directors of Resin Research Laboratories, Inc., Newark, New Jersey.

**Raymond C. Adams** of Gilman Paint & Varnish Co., Chattanooga, was named president-elect of the Federation of Paint and Varnish Production Clubs. **Howard G. Scholl** of C. M. Athey Paint Co., Baltimore, was installed as the 37th president of the Federation.

**Gerald F. Delaney** has been appointed to the Platecoil Division of Tranter Mfg., Inc., Lansing, Mich.



Cathodic protection supplies in new Cleveland warehouse.

## ANNOUNCEMENT! CSI OPENS CLEVELAND, OHIO PLANT

Here's another reason why you'll find it pays to check with CSI for help in preventing corrosion on buried metal structures of all kinds. Now, to serve you faster and better, CSI has opened a new office and warehouse at 1309 Washington Ave., Cleveland 13, Ohio.

CSI engineers are pioneers and experts in the cathodic protection of transmission and distribution pipelines, oil and gas well casing, tank bottoms, etc. They offer a complete line of:

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  - Good-All rectifiers to meet your requirements
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  - Instruments by Agra, Fisher, Collins, etc.
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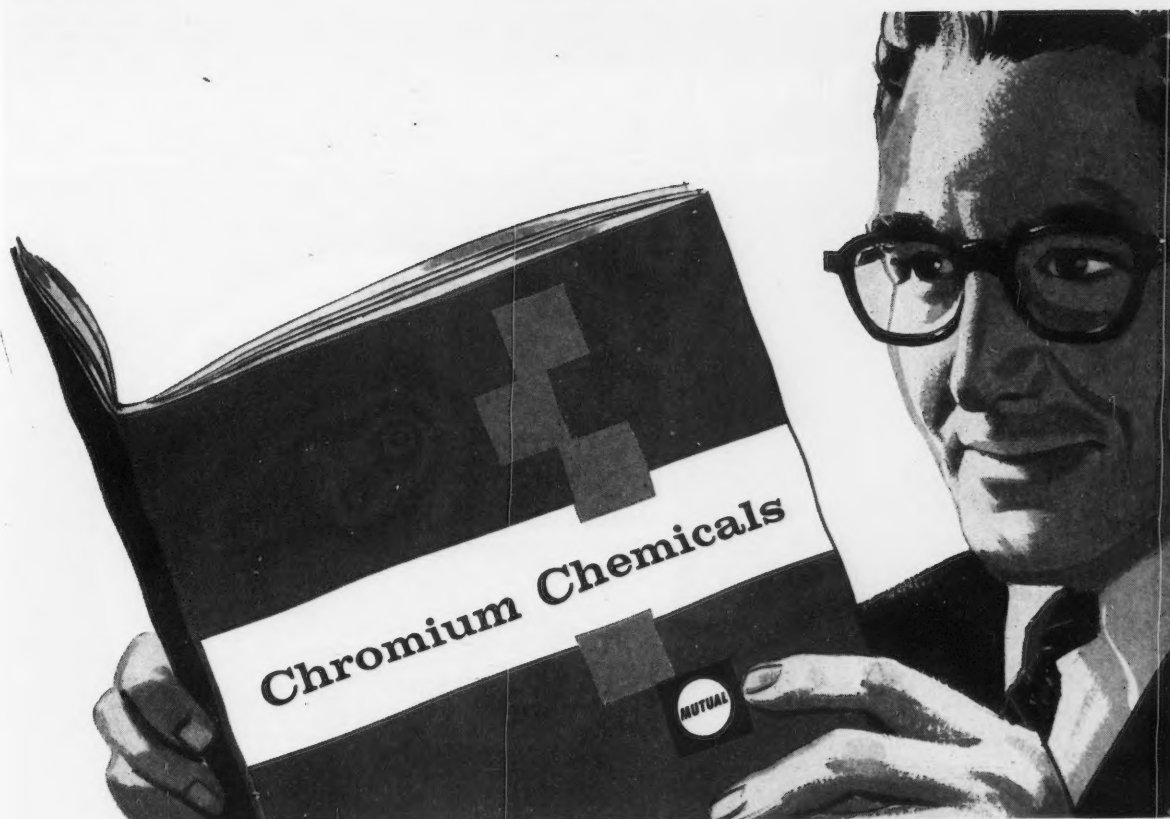
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Anhydrous		X	X		X	X	X			
Tetrahydrate					X	X			X	
CP		X			X					
Sodium Bichromate					X	X	X		X	X
Technical*					X	X				
CP					X					
Potassium Bichromate					X	X			X	X
Technical					X	X				
Granular					X	X				
Powdered					X					
CP		X								
Potassium Chromate		X			X					
Technical		X			X					
CP		X			X					
Ammonium Bichromate					X					
Technical		X			X					
CP		X			X					
Chromic Acid						X	X			
Koreon "M" & "X"				X				X		

\* Also shipped as 69% solution in 8000-gallon tank cars.

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Chromic Acid

Potassium Bichromate  
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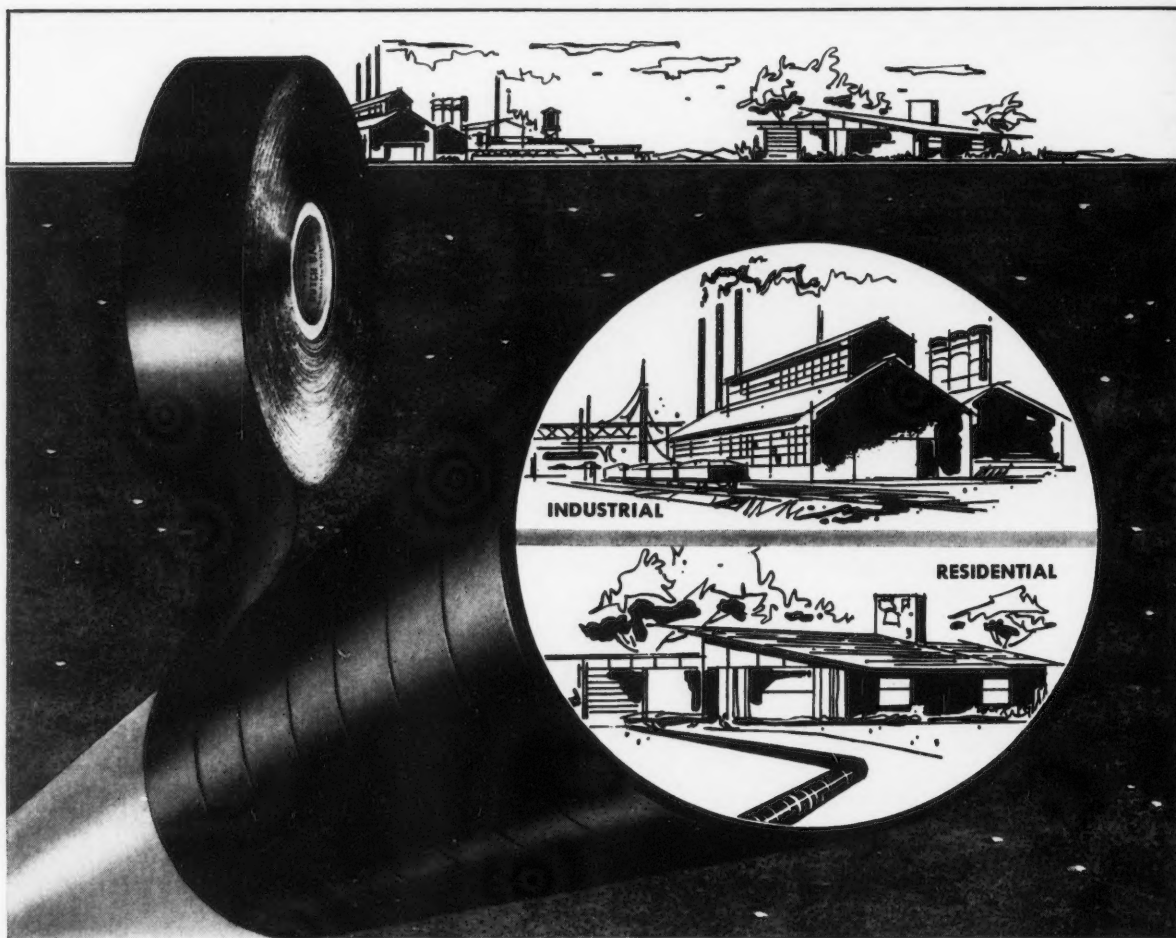
- ☐ Bulletin 52—Chromium Chemicals
- ☐ Bulletin 55—Corrosion Inhibition with Chromates in the Oil and Gas Industries.
- ☐ Bulletin 53—Corrosion Inhibitors in Recirculating Water Systems.
- ☐ Bulletin 35—Corrosion Control in Air Conditioning.

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rents. Because of a patented Johns-Manville bonding process, Trantex always sticks tight . . . won't be budged by underground pressures . . . resists abrasion and penetration, air leakage or chemicals in solution. Put Trantex to the test. See for yourself how Trantex fights costly corrosion! Johns-Manville Dutch Brand Division, 7800 South Woodlawn Ave., Chicago 19, Illinois.



**JOHNS-MANVILLE**





## TECHNICAL TOPICS

four case histories

# Intergranular Corrosion Of Austenitic Stainless Steel<sup>\*</sup> in petroleum refineries

### Abstract

Four case histories of intergranular corrosion of austenitic stainless steels in a petroleum refinery are given. Each considers special applications, analysis of causes and solutions of problems. Cases considered include Type 304 furnace tubes in a thermal cracking plant, Type 316 pipe lines in a fixed bed catalytic reforming unit, Type 316 linings in pressure vessels handling phosphoric acid and Type 316 vessel containing organic acids.

3.2.2

**A**LTHOUGH LATE developments in the manufacture of austenitic stainless steel have lessened this material's susceptibility to corrosion by lowering the carbon content of steel, special applications of stainless steel have produced new corrosion problems. Four case histories of intergranular corrosion encountered with stainless steel in a petroleum refinery illustrate the recurring corrosion problems facing industry. The cases considered are:

Type 304 steel furnace tubes in a thermal cracking plant.

Type 316 pipe lines in a fixed bed catalytic reforming unit.

Type 316 linings in pressure vessels handling phosphoric acid.

Type 316 vessel containing organic acids.

### Type 304 Steel Furnace Tubes

In thermal cracking plants in which high sulfur stocks are processed, Type 304 steel has proved to be desirable for furnace tubes. Tubes in this service showed no intergranular corrosion after operating up to seven years. However, after this furnace had been shut down for six months and was being prepared for further operation, several tubes ruptured during hydrostatic tests. Examination of the failed tubes showed that intergranular corrosion had attacked both the inner and outer walls.

Internal corrosion was confined to the bottoms of the tubes where water had collected while the furnace was not in operation. This water had become acidified either by absorption of sulfur dioxide from the surrounding atmosphere or by oxidation from iron sulfide scale, thus producing a weak sulfurous acid solution which caused the corrosion.

Unlike the internal corrosion that affected only the bottom areas of the tubes, the external corrosion was uniform. This latter corrosion was caused by moisture absorption of the deliquescent salts in the ash deposits on the tubes. Deterioration of workmen's overalls after furnace inspections proved that the moisture deposits on the tubes were strongly acidic.

Corrosion in furnaces containing Type 304 tubes which are to be out of use longer than the usual shut-down period has been eliminated by filling the tubes with oil and by sandblasting and oiling their exterior surfaces.

### Type 316 Pipe Lines

The Type 316 outlet line in the hydrocarbon system of a fixed bed catalytic reforming unit developed a crack along the bottom near a motor-controlled gate valve. During operation, this line reached temperatures as high as 1000 F. Periodic inspections of the external surfaces over a period of several years had shown no signs of cracking or corrosion.

When the cracked section was removed and examined, minor pitting was found on the internal bottom area extending several feet from the valve. Sectional cuts adjacent to the crack revealed general intergranular corrosion on the interior of the line and heavy intergranular corrosion in local areas. Severity of the attack on local areas presumably was influenced by stress.

Further examination showed that sealing steam leaking through badly scored valve seats caused the corrosion after it had combined with sulfur dioxide regeneration gases in the line.

This corrosion problem was solved by using a dry inert gas instead of steam for the valve sealant. No further corrosion

has been discovered after ten years' operation using the inert gas.

### Type 316 Vessel Linings

Intergranular failures have been encountered in Type 316 sheet lines pressure vessels handling hot concentrated phosphoric acid. The acid attacked the mildly sensitive areas where the steel linings had been welded. Welded joints for even 1/8-inch sheets proved to be sensitized sufficiently for corrosion to occur. The attack is characterized by a granular or sugary appearance in the heat-affected zone adjacent to welds.

Although some improvement was attained by careful regulation of welding procedures, life of the vessel linings has been substantially lengthened through use of Type 316L stainless steel. Even with Type 316L, however, welding must be done with care to limit the amount of heat applied if the carbon content of the steel is not below 0.025 percent.

### Type 316 Vessel Containing Organic Acids

Since aqueous solutions of organic acids similar to maleic had been handled in regular Type 316 vessels for several years, a 14 by 30-ft. vessel was built of 1/4-inch Type 316 steel for the processing of maleic acid at 160 F. Because the chloride content of the water was considered to be high, the finished vessel was stress-relieved at 1550 F. It was air cooled because its size made rapid cooling impossible.

After three months' service, examination of the vessel showed large areas that were corroding intergranularly. After six months, several places near welded seams were penetrated. Specimens of Type 316L steel, which had been welded to the vessel after being stress-relieved and slowly cooled, showed no sign of corrosion attack after six months' exposure in the acid.

The original vessel was replaced with a Type 316L vessel which has shown no intergranular corrosion after a year.

<sup>\*</sup> Adapted from a paper "Resume of Intergranular Corrosion of Austenitic Stainless Steels" by J. R. Cunningham, Standard Oil of California, Richmond, Cal., given at a meeting of Western Region, National Association of Corrosion Engineers, San Diego, Cal., October 23-25, 1957.

**WHAT ARE** engineering colleges doing toward training corrosion engineers? This question has several implications. Does it mean turning out graduates with the label Corrosion Engineer at the end of 4 years? Or does it mean basic engineering training upon which the young engineer may build to become a specialist in solving corrosion problems?

If a BS in corrosion engineering is contemplated, what would be the major subjects, the complex chemistry for inhibitors, the electrical engineering and physics necessary for cathodic protection, the metallurgical specialist or the specialist in design? All these things have a definite relation to the problems of corrosion mitigation. The preliminary preparation for any one of these specialties will crowd a four-year curriculum, and the specific preparation for each specialty will be quite different.

The specialist in inhibitors may require the basic training of a chemical engineer or a chemistry major with a considerable amount of additional study in physical chemistry and biology. Usually he will not have the opportunity for more than a very superficial introduction to electric current phenomena, metallurgy or design.

The specialist in cathodic protection would require the preliminary training of the electrical engineer with additional work in electro-chemistry and electrical measurements. His undergraduate work in chemistry, metallurgy and engineering design would of necessity be superficial.

Perhaps a better method would be to train engineers who may choose to go into some phase of corrosion control work. The most direct approach for preliminary preparation is through the fields of chemical, electrical, metallurgical or mechanical engineering.

The thinking of some engineering educators expressed in the following quotations may indicate the philosophy and objectives of undergraduate engineering training.

A dean of engineering stated, "I do not consider it educationally wise to offer a course in such a detailed field in the undergraduate level. Our objective in engineering education at this institution is to attempt to enhance the later ability to learn rapidly in the technological field and to create a zeal for continuing education."

#### Four Year vs Five Year Course

A department head in chemical engineering says "In general I think corrosion is an important subject but on the other hand so are several others. It is a question of time. How much can you crowd into four years or what should be taken out? . . . I personally am against a required 5 year course for a BS degree. There is a continual demand for more mathematics and some cultural courses. There is also the desire of many groups to have more time devoted to their specialty, for example, nuclear technology, instrumentation, paint technology or vegetable oil technology. It is my feeling that a four year course in one of the main branches of engineering with emphasis on the fundamentals is best and not to have fragmentation into specialties. Specialization could be handled by graduate work or

# Corrosion Control Courses

## What Colleges Are Teaching\*

### Abstract

This paper discusses the time limitations on four-year engineering curricula as to course content. It points out that in practically all engineering curricula some reference is made to the principles of corrosion in basic science courses. In many schools some emphasis is given to the subject of corrosion in courses that do not include the word in the title. Thus it is safe to assume that practically all graduates of a four year curriculum have had some exposure to the subject.

The 85 replies to a questionnaire sent to 103 engineering colleges indicate an increasing number of formal courses on corrosion offered at the graduate level and also an increasing interest in research by both faculty and graduate students. 1.1

perhaps by training within a company organization."

The opposition to a 5-year program leading to a BS is rather general. The engineering graduate is already completing 145 or more semester hours of work while the liberal arts graduate is required to do only 124. Since many companies pay a premium for advanced degrees, this puts the engineering graduate at a disadvantage. In many cases he is doing almost as much work for his bachelors as the Arts and Sciences graduate does for his masters.

### Engineering Curricula

The Engineering Council for Professional Development is the accrediting agency for engineering curriculums. Its criteria for accreditation states that an approved engineering program must contain at least the following equivalents:

1 year of basic sciences (mathematics, physics, chemistry). Note that one year in engineering usually means 36 semester hours.

1 year of engineering sciences presenting applied mechanics, strength of materials, fluid mechanics, thermodynamics, transfer and rate mechanisms (heat mass momentum), electrical theory and nature and properties of materials.

½ year minimum (1 year desired) for humanistic and social studies, exclusive of English composition, speech, accounting, drawing, management, personnel management and ROTC. (These excluded subjects are requirements in most engineering curricula and amount to a minimum of about 31 semester hours.)

½ year devoted to engineering analysis, design and engineering systems.

Thus 3 to 3½ years of work are definitely allocated with practically no opportunity for specialization. The subject matter excluded under the item Humanistic and Social Studies amounts to al-

most the equivalent of one school year. Consequently the undergraduate is asked to crowd up to 4½ years of requirements into a 4-year program. Many readers will remember their own college experience as being crowded and rather rugged.

To have an active major curriculum, there must be student demand. In general the enrollments have been small in the corrosion courses.

### Questionnaire Results

This rather lengthy introduction is not to alibi for what the schools are not doing but rather to explain why they are doing as they are. A questionnaire was sent to 103 colleges having four year programs in engineering. Replies were received from 85 schools. Several replied by writing letters explaining their offerings more fully than could be done by filling the blanks on the questionnaire.

In practically every engineering curriculum there is some discussion of corrosion in one or more courses required of every engineering student. In beginning chemistry, corrosion is taught under oxidation; in physics, galvanic currents are measured and the galvanic series of metals is usually introduced. In basic electrical courses dealing with batteries, galvanic action is also discussed. In engineering materials courses, several class periods are usually devoted to general principles of corrosion and corrosion resistance of various metals and alloys.

Seventy of the schools listed one or more courses in which a discussion of corrosion is an important part. Most of the courses mentioned were in the fields of chemical engineering, metallurgy and metallurgical engineering. Others listed mechanical engineering design, engineering materials and electro-chemistry.

Thirty-five schools listed specific courses in corrosion. Most of these courses are for senior or graduate students. Where these are listed as elective undergraduate courses, they are infrequently taught because of lack of student demand. The first formal course in corrosion listed in the replies was offered at MIT about 1930. There probably were some courses in existence before that time. Of the schools listing these corrosion courses, 30 of them started the courses in 1947 or later. Most significant growth in corrosion courses occurred during the 1947-50 and 1956-57 periods.

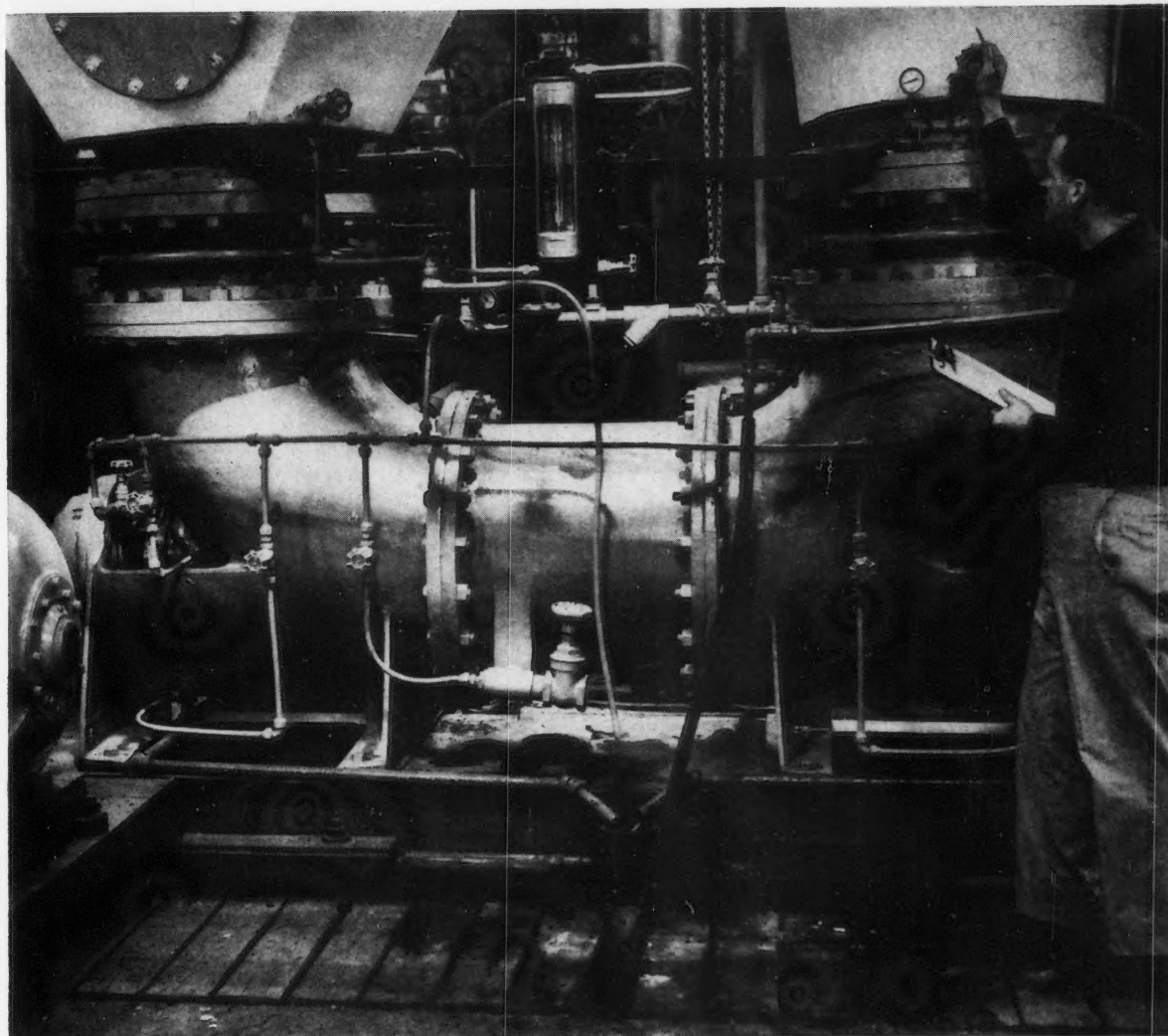
### Short Courses

Twelve schools sponsor or co-sponsor short courses. Two others are planning to sponsor such courses in the near future. These short courses vary in length from the 12-week night course started in November, 1956, at Rennselaer Technical Institute to the more usual 3-day session participated in by both schools and industry. In the latter type the papers are predominantly from men in industry. The first short course jointly sponsored by NACE was at the University of Texas in 1942. Most of these programs have been conducted since 1950.

The sentiments expressed by those who have conducted short courses ranged from enthusiastic to "never again." In general the feeling seems to be that the short course is a good method for arousing interest in the subject and at the same time spreading

(Continued on Page 124)

\* A paper presented by R. V. James, Professor Mechanical, University of Oklahoma, at a meeting of the South Central Region, National Association of Corrosion Engineers, Oklahoma City, October 1-4, 1957.



One of two new axial flow pumps made for the International Salt Company by Morris Machine Works of Baldwinsville, N. Y. This 24-inch, Type 316 stainless pump recirculates 14,000 gallons of salt slurry per minute through an evaporator.

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## Corrosion Courses—

(Continued From Page 122)

information on the processes and prevention of corrosion.

As pointed out earlier, corrosion courses and the serious study of corrosion find their natural place in the graduate curriculum. Twenty-seven schools have active research programs. Twenty-one research programs are for graduate students. The staff is also active in research at 21 institutions while 16 schools have co-operative or contract programs. Several of these schools have all three types of research in progress.

At 42 schools the staff members have published articles and technical papers on corrosion and have presented lectures on the subject.

From the results of this survey a considerable and growing interest in the subject of corrosion is evident. Engineering colleges are keeping step with this growing interest by providing opportunities for study at the appropriate levels. This increased interest closely parallels the realization by industry of the importance and tremendous cost of corrosion and the increasing desire to do something about it. It is reasonable to suppose that the study opportunities in engineering colleges will continue to grow with the increasing emphasis by industry on corrosion problems.

### Summary

The answer to the question "what are engineering colleges doing toward training corrosion engineers?" may be summarized as follows:

Practically all engineers are given a brief introduction to the principles of corrosion at the undergraduate level.

Comprehensive study of corrosion and corrosion problems can be done at the graduate level in many engineering colleges.

Few undergraduate colleges will offer degrees in corrosion engineering in the near future.

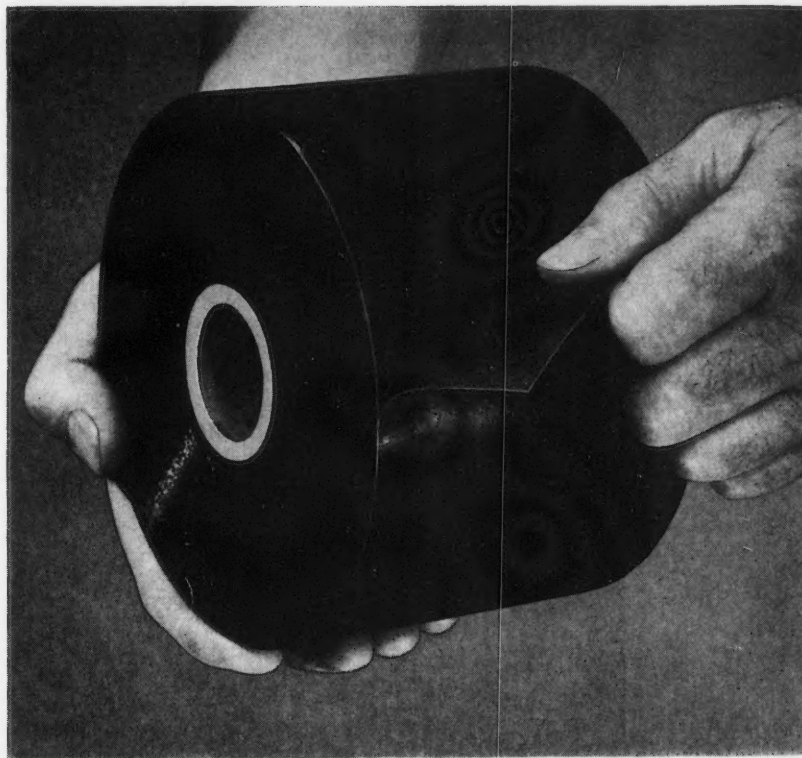
Short courses are available in all parts of the United States for refresher courses and to supplement on the job training for those actively engaged in corrosion mitigation.

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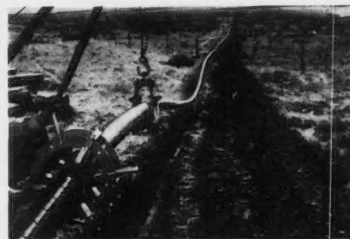
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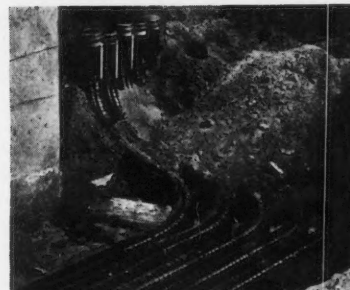
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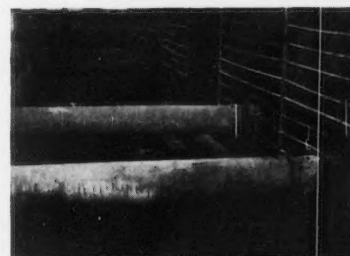
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**sodium hypochlorite**  
**solution**  
**(reagent quality)**







# CORROSION ABSTRACTS

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### 3. CHARACTERISTIC CORROSION PHENOMENA

#### 3.2 Forms

3.2.2, 7.6.4, 5.8.2

Intercrystalline Corrosion Called "Caustic Cracking", in Steam Boilers. (In Spanish.) MANUAL ADROE IGLESIAS AND JOSE M. HAUSMANN MORO. *Acero y Energia*, 14, 35 + 14 pp. (1957) July-August.

Historical background of caustic cracking. Description of the cracks. Location and identification. Use of a Schroeder detector. Inhibition of the phenomenon. Chemical inhibitors: Sodium sulfate, sodium orthophosphate, sodium nitrate and tannins. Analytical methods.—BTR. 15328

3.2.2, 8.9.5

An Investigation of Cracked A-212 Catapult Steam Receivers. A. J. BA-  
BECKI AND P. P. PUZAK. U. S. Navy  
Dept., NRL Report No. 5058, Dec. 10,  
1957, 12 pp.

Base metal cracking in vicinity of nozzle and manhole reinforcement pads was discovered recently in several 65-inch diameter catapult steam receivers aboard 4 naval aircraft carriers after 1-1/2 years of service. Cracking was evident only on inside surface of receivers, generally following toe of filler weld between shell and reinforcement pad. Fabrication defects in form of porosity, undercutting and poor fit-up were found in all 3 receiver sections. Normal oxidation scale on inside receiver surfaces was similar to that found on crack surfaces; however, operating

condition were such as to preclude requirement of unusual corrosive conditions for cracking. Typical fatigue-type markings of crack propagation were found on crack surfaces emanating from weld-metal porosity. Microscopic examination disclosed cracks to be wholly transgranular; slow propagation of fissures was indicated by heavily deformed metal contiguous to line of propagation. Brittle fracture was not involved. Fabrication defects and stresses resulting from design particulars are believed to be principal causes for development of these premature fatigue failures under normal accumulator service conditions. Compositions given of shell, reinforcing pad and weld metal include nickel content. Photomicrographs, tables, graphs, diagrams, photos.—INCO. 15243

3.2.2, 3.7.3, 3.5.9, 6.2.3

Microcracking in Mild Steel Weld Metal. K. WINTERTON. *Can. Metalworking*, 20, Nos. 9, 10; 34, 36, 38-40, 42; 58-60, 62, 64 (1957) Sept., Oct.

Rapid cooling rates associated with welding at low temperatures give microcracking special importance. Phenomenon of microcracking in metal-arc, mild-steel weld-metal is concerned with formation of large numbers of small cracks in the weld metal from most types of electrodes under conditions of rapid cooling. Considerably less microcracking was found with low hydrogen basic-coated types. A general theory is put forward to account for microcracking in terms of accumulation of hydrogen under pressure in pre-existent flaws. Effect of relatively small deviations in plate temperatures on low temperature cooling is quite pronounced. Non-metallic inclusions found in mild steel weld metal are classified into 5 general types. Any of these can be associated with microcracks, but most important seem to be rhodonite inclusions containing free silica. Tables, photomicrographs, graphs, 19 references.—INCO. 15155

3.2.2, 6.2.5, 7.2

The Effect of Localized Carburization on the Corrosion Resistance of Stainless Steel Pipe and Tubing. WILLIAM L. WALKER. General Electric Co. U. S. Atomic Energy Commission Pubn., HW-51267, July 8, 1957, 7 pp. Available from Office of Technical Services, Washington, D. C.

Three small-diameter 304L stainless steel pipes which failed the Huey test were examined. It was found that the greater weight loss occurred inside the tube and high corrosion rates were observed until the carburized layer was removed. This layer is said to have resulted from incomplete cleaning prior to heat treatment.—NSA. 15133

3.2.2, 6.3.15

The Influence of Hydrogen on Delayed Failure in Titanium Alloys. R. D. DANIELS, E. L. HARMON, JR. AND A. R. TROIANO. Case Institute of Technology. U. S. Wright Air Development Center, Tech. Rept. 57-30, Feb., 1957, 41 pp.

Notch geometry, strength level, hydrogen concentration and hydrogen distribution exerted an appreciable influence

on delayed failure characteristics of a 4 aluminum-4 manganese, titanium alloy. Hydrogen-induced delayed failure in sharply notched material occurred by a process of crack initiation and slow crack propagation. Average rate of crack growth was accelerated as hydrogen content increased and appeared to be independent of applied stress. Crack initiation and propagation process under certain conditions of stress was complicated by room temperature creep. A high concentration of hydrogen at surface of a specimen was conducive to early crack formation; and crack growth rates appeared dependent upon inward diffusion of hydrogen from surface. Graphs, photomicrographs, diagrams.—INCO. 13767

### 3.8 Miscellaneous Principles

3.8.2

The Electrochemical Properties of the Stressed Layer of the Surface of Pure Iron. N. OHTANI. *J. Japan Inst. Metals*, 20, No. 7, 400-403 (1956) July.

Assuming that the corrosion of pure iron is controlled equally by anode and cathode reactions, the electrochemical properties of the stressed layer of the

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surfaces of specimens polished by various methods, that is, the order of the variation of the thickness, the electrode potential of the layer and the free energy of the activation of hydrogen electrode reaction were studied.—JSPS.

13309

## 3.8.2

**Corrosion as an Electrochemical Process.** (In English.) HERBERT H. UHLIG. *Electrochem. Soc., Bulletin of India Section*, 5, No. 1, 11-14 (1956) Jan.

Theoretical considerations. Graphs, tables. 3 references.—BTR.

12630

## 3.8.3, 6.3.3

**Anodic Passivation of Chromium in Acid Solutions.** (In Russian.) V. M. KNIAZHEVA AND IA. M. KOLOTYRKIN. *Doklady Akademii Nauk SSSR*, 114, No. 6, 1265-1268 (1957) June 21.

The electrochemical behavior of pure chromium was studied by the potentiometric method during its passivation in 0.01 and 0.10 and 1.0 N solutions of sulfuric acid.—BTR.

14972

## 3.8.4

**Oxidation of Alloys Containing Metallic Compound NiAl.** (In Russian.) E. I. MOZHUKHIN, L. KH. PIVOVAROV, AND IA. S. UMANSKII. *J. Applied Chem. USSR (Z. Prikladnoi Khim.)*, 30, No. 11, 1593-1599 (1957) Nov.

Heat-resisting qualities of the single-phase alloys composed of the NiAl only, and of the two-phase alloy NiAl + Ni<sub>3</sub>Al. Oxidation of NiAl depends on composition; an excess of aluminum atoms produced a denser oxide film.—BTR.

15378

## 3.8.4, 4.3.2, 6.3.21

**The Oxidation of Mercury by Nitric Acid.** R. F. MURRAY AND H. SCHNEIDER. Phillips Petroleum Co. U. S. Atomic Energy Commission Pubn., IDO-14423, Nov. 30, 1957, 24 pp. Available from Office of Technical Services Washington, D. C.

The oxidation of mercury in nitric acid was investigated. An empirical rate law, oxidation rates and mercury species in solution were determined. A reaction mechanism based on the presence of nitrous acid can be interpreted in terms of the rate expression derived. (auth.)—NSA.

15377

## 3.8.4, 3.7.2, 6.4.3

**Iron as a Corrosion Inhibitor in Beryllium with Notes on Vectorial Corrosion in Single Crystals.** C. S. PEARSALL. Massachusetts Institute of Technology. U. S. Atomic Energy Comm. Pubn., MIT-1109, April, 1953 (Declassified Feb. 25, 1957), 19 pp. Available from Office of Technical Services, Washington, D. C.

The vectorial nature of corrosion in beryllium single crystals was studied. The effect of aluminum, iron, and silicon impurities on the corrosion behavior of beryllium is reported.—NSA.

14750

## 3.8.4, 3.4.9

**Metal-Water Reactions. Pt. IV. Kinetics of the Reaction Between Calcium and Water Vapor.** HARRY J. SVEC AND CHARLES APEL. Iowa State College. *J. Electrochem. Soc.*, 104, 346-349 (1957) June.

Calcium metal was reacted with water vapor in the temperature range 20 to 70°C and at water vapor pressures of 18 to 93 mm. Hg. The experiments indicated that the only products were calcium hydroxide and hydrogen and that the reaction proceeded according to the

equation  $\text{Ca} + 2\text{H}_2\text{O} \rightarrow \text{Ca}(\text{OH})_2 + \text{H}_2$ . A manometric study of the reaction showed that it followed the logarithmic rate law. The rate constant was observed to be linearly dependent on water vapor pressure below 70°C and to decrease with increasing temperature in the range 20 to 50°C. At 70°C the rate constant was observed to be independent of water vapor pressure. The activation energy was found to be -7.52 kcal/mole in the pressure dependent range. (auth.)—NSA.

14751

## 3.8.4, 6.4.4

**Phase Relationships in Magnesium Alloys.** J. J. PARK AND L. L. WYMAN. National Bureau of Standards, U. S. Wright Air Development Center, U. S. Air Force, October, 1957, 33 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. (Order PB 131622.)

This final report summarizes results and conclusions of a project with two objectives. The first was establishment of the solubility of lanthanide metals in magnesium and the pertinent conditions of equilibria. The second was to establish the solubility of zinc in magnesium and to determine the phase or phases with which the magnesium terminal solid-solution is in equilibrium. Phase relationships in magnesium-base alloys were studied by thermal, X-ray and metallographic methods. The magnesium-rich portions of the binary phase diagrams of magnesium plus the rare earth metals lanthanum, cerium, praseodymium and neodymium were determined. A thorough investigation of the magnesium-zinc system from zero to more than 66.67% zinc was also conducted and the presence of four intermediate phases with their solubility ranges was established. In addition, the extent of the solid solubility of zinc in magnesium was determined.—OTS.

15389

## 4. CORROSIVE ENVIRONMENTS

## 4.3 Chemicals, Inorganic

## 4.3.2, 6.3.6

**Rate of Copper Oxidation in Nitric Acid.** (In Russian.) N. N. MILYUTIN AND A. I. SHULTIN. *J. Applied Chem., USSR (Zhur. Priklad. Khim.)*, 30, 58-62 (1957) Jan.

Oxidation rates in nitric acid of various concentrations is expressed quantitatively by the equation:  $\phi = 0.195 + 0.056 \ln v$ . Increase of temperature accelerates the oxidation process.—BTR.

14338

## 4.3.2, 6.3.21, 6.6.6

**Nitric Acid Dissolution of Thorium. Kinetics of Fluoride-Catalyzed Reaction.** ROBERT LEE MOORE, C. A. GOODALL, J. L. HEPWORTH AND R. A. WATTS, JR. Hanford Atomic Products Operation. *Ind. and Eng. Chem.*, 49, 885-887 (1957) May.

The penetration rate of thorium metal by boiling nitric acid was measured as a function of the concentrations of acid, fluoride catalyst, and dissolved thorium. The fluoride concentration was the most important variable, the dissolving rate in 13M nitric acid increasing from 1 mg per hour per sq cm in the absence of fluoride to over 10,000 mg per hour per sq cm at 0.1M fluoride. The rate also increased with increasing nitric acid and reached a maximum at about 13M. Dissolved thorium inhibited the reaction, presumably by complexing the free fluo-

ride. Aluminum had a similar effect. The dissolution rate of thorium was found to be much less than that of thorium. "Integral" dissolvings were also made, and an empirical mathematical equation was derived to correlate these results and facilitate application. (auth.)—NSA.

## 4.3.2

**The Behaviour of Materials in Anhydrides and Aqueous Hydrofluoric Acid.** E. LINGNAU. *Werkstoffe und Korrosion*, 8, 216-233 (1957) April.

Review of literature on behavior of materials in anhydrides and aqueous hydrofluoric acid and hydrofluoric acid gas. Data are given on steel, high- and low-alloy nickel-chromium, chromium and nickel-chromium-molybdenum steels, cast steels, Carpenter 20, Cooper V2B, Durimet 20, Monel, nickel, Hastelloy B, Chlorimet 2, Inconel, Hastelloy D, copper, silicon bronze, cupro-nickels, silver, Ilium R, welded joints of the above materials, aluminum, magnesium, platinum metals, titanium, niobium, tantalum, molybdenum and non-metallic materials. Recommendations as to suitable applications of the various materials are included.—INCO.

14312

## 4.3.2, 5.9.2, 5.8.2

**Inhibitors for Acids.** J. TOMISLAV AND S. MILANKA. *Zastita Materijala*, 5, No. 6, 201-203 (1957).

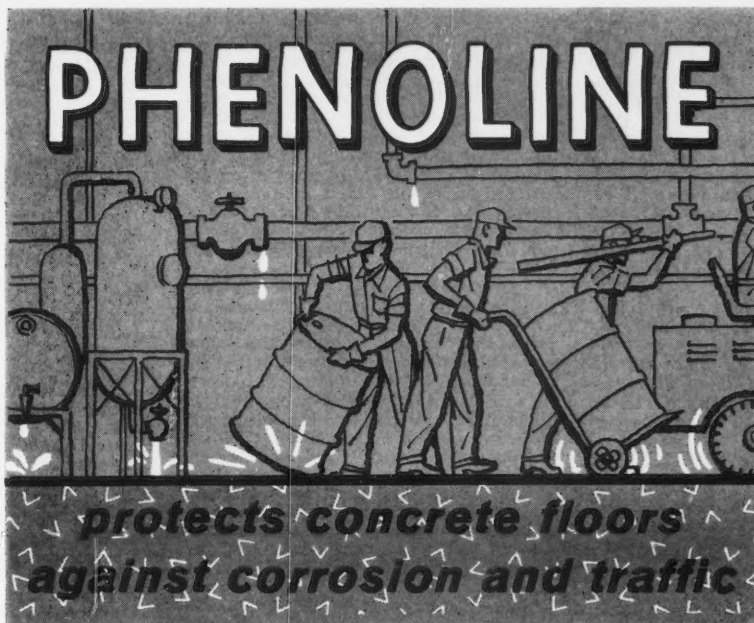
The article describes the classification of inhibitors for acids, their importance and the mechanism of their activity. The efficiency of various inhibitors for the pickling of steel in sulfuric acid and the effect of their concentration have been examined. The speed of corrosion in sulfuric acid has been measured with and without inhibitors, and on the basis of the data obtained the inhibiting capacity has been calculated. Various organic compounds which contain nitrogen and sulfur have proved to be the most efficient, e.g., o-cresylthiourea and phenylthiourea.—RPI.

14791

## 4.3.2, 5.8.2

**Inhibiting Effect of Hydrofluoric Acid in Fuming Nitric Acid on Liquid and Gas Phase Corrosion of Several Metals.** DAVID M. MASON, LOIS L. TAYLOR AND JOHN B. RITTENHOUSE. *Corrosion*, 13, No. 12, 821t-827t (1957) Dec.

Measurements were made at 130 F of the extent of corrosion of several metals exposed to the liquid and the gas phase of thermally stable nitric acid containing 11 to 13 weight percent nitrogen dioxide and 2 to 4 weight percent water with and without hydrofluoric acid added as a corrosion inhibitor. Liquid-phase corrosion of the following metals was readily inhibited by hydrofluoric acid in fuming nitric acid of this composition: aluminum alloys 2S-O (1100), 14S-T6 (2014-T6), 17S-T4 (2017-T4), 24S-T4 (2024-T4), 61S-T6 (6061-T6), and 75S-T6 (7075-T6); aluminum 2S-O welded to 356; and chromium-nickel steels 302, 303, 304, 321, 347, Armco 17-7PH, and Uniloy 19-9DL and 19-9DX. Carbon steel C1020 and chromium alloy steels 4130, 410, 430 and 446 having intact natural metal oxide films, which were formed in moist air, were inhibited by hydrofluoric acid. Corrosive attack of these steels without the oxide film, however, was aggravated by the presence of hydrofluoric acid in fuming nitric acid, the corrosion becoming more extensive the lower the chromium content of the steel. Commercially pure lead was inhibited by hydrofluoric acid,



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For severe chemical conditions, but little trucking or other heavy traffic: e.g., beneath tanks and equipment.

**SYSTEM:** Prime coat—Phenoline 300 Orange  
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Top coat—Phenoline 300  
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whereas pure chromium, Nickel-A, tantalum, and titanium alloys 75A and 130A were either only slightly inhibited or showed corrosion rates which actually were increased by presence of hydrofluoric acid in fuming nitric acid. Inhibition of gas-phase corrosion was found to occur readily in the case of the following metals tested: steels 303, 410, 430, and 446 and aluminum 61S-T6. Gas-phase corrosion of steels 4130 and 1020 was usually aggravated by hydrofluoric acid. Exposure of aluminum 61S-T6 and stainless steel 347 to fuming nitric acid with a repeated cycling of temperature between 70 and 160 F was found not to impair the inhibiting effect of hydrofluoric acid on gas and liquid-phase corrosion of these metals.

A few tests of the liquid and gas-phase corrosion of aluminum 61S-T6 and steels C1020 and 347 by nitrogen dioxide at 130 F were also made, and this medium was found in general to be much less corrosive than fuming nitric acid. 14446

## 5. PREVENTIVE MEASURES

### 5.1 General

#### 5.10, 3.2.2, 6.2.3

**The Arrest of Brittle Cracks in Ship Plate.** J. HUNTER. Paper before Australian Welding Inst., 4th Ann. Conv., Brisbane, October 11, 1956. *Australasian Engr.*, 1957, 52-58, Feb. 7.

Series of tests, using method developed by Admiralty in United Kingdom and described by T. S. Robertson, indicates possibility of arresting low temperature brittle cracks in ship plates by introducing narrow strip of austenitic stainless steel or in some applications by transverse butt weld of austenitic stainless steel. All plates tested were 15 lb. ( $\frac{3}{8}$ " ) ship plates; insert plates were commercial quality 18-8; and stainless steel electrodes were commercial quality fully extruded 25 chromium-20 nickel with lime type covering. Appendix covers single tests on mild steel ship plate showing that composite ferritic-austenitic transverse butt weld arrested a brittle crack, while transverse surface beads of austenitic weld metal did not arrest crack.—INCO. 13815

#### 5.11

**Factors in the Selection of Corrosion Resistant Materials.** F. L. WHITNEY, JR. Monsanto Chem. Co. *Metal Progress*, 71, No. 6, 90-96 (1957) June.

Three major factors which should be considered in selection of a metal for corrosive service are material, environment and economics. Importance of selection of suitable material is shown by description of behavior of stainless steels, lead, chromium-nickel-molybdenum-tungsten alloy and Hastelloy in different applications. Table gives corrosion rates for reaction vessels of Everdur, Type 304, Monel and steel. Ease of fabrication, corrosion rate and cost are most important items, but operating factors—such as temperature, concentration, purity and velocity of corrosive fluid, erosion by sludge, concentration cells under adhering solids—are of equal influence on the over-all economics. Photomicrographs, graphs, photos.—INCO. 14383

#### 5.11

**Corrosion Prevention Starts with Design.** H. P. GODARD. *Ind. and Eng. Chem.*, 49, No. 11, 79A-81A (1957) Nov.

206a

Dr. Godard states that "the majority of corrosion complaints encountered in equipment service are due to improper choice of alloy, poor design and faulty construction." He illustrates this dictum by citing several examples. One of these concerns high-voltage electrical transformers in an aluminum reduction plant where, in 1952, two transformers in a group of 21, failed in quick succession due to corrosion perforation of their copper cooling coils after only a moderate period of service. This failure led to a series of failures in other parts of the plant as a direct consequence. The remedy, treatment of the coolant water, was simple, but attention to corrosion during design would have provided for water treatment and the failure would not have occurred. Knowledge of corrosion rates is essential in predicting equipment life. However, these can be misleading unless the conditions under which they were determined are fully described and are similar to the case in question. Corrosion rates are influenced by physical factors of the environment such as temperature, velocity, turbulence of agitation and the presence of entrained solids. Traces of impurities also have an important influence.—ALL. 14885

#### 5.11, 7.5.1

**Strength of Cylinders.** W. R. D. MANNING. *Ind. and Eng. Chem.*, 49, No. 12, 1969-1978 (1957) Dec.

Chemical industry has greatly increased importance of correct design for pressure containers. Combined effects of such factors as corrosion, high temperatures and fluctuating loads have raised demands beyond those which can be met by straightforward interpretation of ordinary theoretical methods. Description is given of new design methods for meeting problems of fatigue and creep imposed by higher temperatures and fluctuating loads on cylinders. Graph shows fatigue of nickel-chromium-molybdenum steel cylinders. Graphs, tables, 26 references.—INCO. 15363

### 5.3 Metallic Coatings

#### 5.3.1

**Aluminizing of Iron and Ferrous Metals.** (In French.) LOUIS GRAND. *Rev. Aluminium*, 34, No. 239, 63-73 (1957) Jan.

The production and properties of dipped aluminum coatings are discussed. To ensure complete elimination of the oxide films between the two metals to be bonded, stringent rules are given for the mechanical and chemical pretreatment of the ferrous metal surface and recommendations are presented concerning ordinary and continuous dipping procedures carried out under a neutral or reducing atmosphere, in a volatile liquid or in the presence of a flux. Surface properties, plasticity and adherence of the aluminum coatings thus obtained are also reported. The advantages obtained by the use of beryllium and R. E. additions, as agents to increase wettability, are said to be questionable. The presence of 2.5 percent to 5 percent silicon in the melt was found to have a beneficial effect in reducing brittleness and exfoliation. Results of corrosion tests carried out with aluminized mild steel in humid atmospheres and at elevated temperatures, are presented.—ALL. 13789

#### 5.3.1

**Sprayed Metal Coatings.** *Corrosion Technology*, 3, No. 10, 311-324, 326-327 (1956) October.

A collection of articles as follows: The Trend of Protective Metal Spraying Practice, W. E. STANTON; Sprayed Aluminum and Zinc in Corrosive Environments, R. E. MANSFORD; Metal Spraying on Clifton Suspension Bridge, R. A. PARKES; Metal Spraying in the Radio Industry, C. F. FISH; Developments in Sprayed Metal Coatings; Metal Spraying at Messrs. Tate and Lyle Ltd., Thames Refinery; Metallised Coatings for Heat Corrosion Protection, R. J. FRANKLIN.—BNF. 13560

#### 5.3.2, 6.3.17

**Iodide Decomposition Process for Coating Uranium with Zirconium.** W. L. ROBB AND F. J. SHIPKO. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-876 (Del.) Feb. 24, 1953 (Declassified with Deletions Feb. 25 (1957), 21 pp. Available from Office of Technical Services, Washington, D. C.

Corrosion-resistant zirconium coatings have been consistently deposited on uranium disks by the de Boer or iodide decomposition process. Four-mil coatings can be deposited in three hours at uranium temperatures of 1090 C and at pressures below one micron. Corrosion tests in boiling water on five disks coated in this way yielded an average time of 263 hours until coating failure. In a flow system which used hydrogen as carrier and reductant for the zirconium iodide, only very thin coatings were obtained. Poor coatings also resulted from attempts to plate with lower uranium temperatures using other metal halides both in vacuum and in a flow system. (auth.)—NSA. 15035

#### 5.3.4

**Aluminum Chromizing: Strength Plus Corrosion-Resistance.** G. R. FULTON AND R. C. REED. *Iron Age*, 179, No. 23, 102-104 (1957) June 6.

Aluminum chromizing differs from conventional chromizing of steel in that it does not decarburize steel and still affords a reliable oxidation and corrosion resistant coating. Welding tests were made to determine weldability of aluminum chromized steel; no weld cracks were produced. Properties of plain carbon steels after aluminum chromizing and of unannealed and annealed aluminum chromized weld specimens are given.—BL. 14964

#### 5.3.4

**Hot-Dipped Aluminum Coatings.** HERBERT L. KEE. *Product Engineering*, 28, No. 17, 57-59 (1957) Oct. 28.

Hot-dipping (in molten aluminum) of steel parts couples the strength of steel to the corrosion resistance properties of aluminum. However, salt-water atmospheres will cause the aluminum to corrode because the coating is anodic to the base metal. The corrosion rate is about four times less than that of zinc. In aggressive atmospheres and dilute oxidizing acids, its corrosion resistance is much greater. Aluminum dipping causes less distortion than other coating processes because: dipping temperatures are between 1300 and 1400 F compared with 1800 and 1900 F; in the hot-dip bath, the weight of parts when suspended in the bath is decreased one-third by the buoyancy of molten aluminum; the molten metal heats the part more rapidly and uniformly. The sus-

pension of parts, post-forming, welding methods for joining treated parts, and brazing methods are covered. To prevent the formation of fillets at the roots of threads, they should be machined after dipping, rather than masked. Centrifuging can be used to clear male threads. For outdoor applications a heavy aluminum overlay (0.005-inch) is preferred. Diffused coatings require a thin overlay (0.001-inch max.) to prevent spalling during diffusion treatment. For optimum ductility, both the alloys and the overlay should be kept to a minimum. Thickness of the intermetallic layer is a function of time, temperature, composition of base metal and bath composition. Overlay thickness, on the other hand, is dependent on drainage of aluminum as parts leave the bath and is more difficult to control than the intermetallic layer. For uniform coating, uniform part-thickness must be maintained.—ALL. 14973

**5.3.4 The Electroplating of Screws and Other Small Articles. II. The Plating Process.** (In German.) WERNER PETERS. *Draht*, 8, No. 2, 40-48 (1957).

Open and immersed barrels are described, methods of charging and emptying are discussed and the relation between current densities mesh-size of immersed barrel, size of screws, rate of revolution and surface finish are considered. Solutions and plating conditions are discussed for bright and dull nickel plating, "electroless" (Kanigen) nickel plating, brass, zinc, cadmium and chromium plating. Finishing treatments such as swelling and drying are considered and the chromate treatment and bright passivating of zinc-plated screws are mentioned.—MA. 15004

## 5.4 Non-metallic Coatings and Paint

**5.4.10, 7.2**

**Polyvinyl Chloride Tape in Corrosion Mitigation Programmes.** A. C. KILBERG. *Corrosion Technology*, 5, No. 1, 10-12 (1958) January.

Discusses properties which make polyvinylchloride tape excellent for electrical application and as a pipe protection tape. Pipe condition for taping, priming properties and machine wrapping methods are described. Photos.—INCO. 15244

**5.4.5, 7.5.5**

**Ballast Tank Protection.** K. V. HODGSON. *Corrosion Prevention and Control*, 4, No. 12, 45-46 (1957) Dec.

Discussion of problem of ballast tank protection. Description is given of development of Marax, a coal tar pitch filming material which possesses phenomenal water resistance and good electrical insulation. Its adhesion to steel is good and therefore a film is produced which is an excellent rust preventive. Marax is very difficult to ignite and does not give off toxic fumes. Full scale trials of tanks on vessel showed them to be in perfect condition after one year. Marax coating gives promise of a serviceable life of several years. Diagram.—INCO. 15313

**5.4.5**

**Painting Steel Drums.** W. A. HAZLETT. *Industrial Finishing*, 33, No. 9, 64-66, 68, 70, 73 (1957).

The hot-rolled steel shells and heads after descaling and alkali, zinc phosphate and chromic acid rinses are as-

sembled and automatically sprayed with an alkyl resin base enamel which is stoved for 15 minutes at 300 F., followed by the printing and marking operations.—RPI. 15298

## 5.8 Inhibitors and Passivators

**5.8.2**

**Sodium Benzoate as Corrosion Inhibitor in Aqueous Dispersions.** (In German.) H. J. FREIER AND W. GEILEN-KIRCHEN. *Werkstoffe u. Korrosion*, 8, 673-677 (1957) November.

Sheet metals intended for containers for aqueous varnish dispersions are protected from corrosion by additives of sodium benzoate, or sodium benzoate-sodium nitrite. Protection depends mainly on acidity. Additions of 2% or less are effective. 9 references.—MR. 15256

**5.8.2, 6.4.2**

**The Use of Minute Amounts of Sodium Dichromate as a Corrosion Inhibitor in Single Pass Aluminum Systems.** D. R. DEHALAS. General Electric Co. U. S. Atomic Energy Commission Pubn., HW-33736, Nov. 11, 1954 (Declassified Jan. 7, 1958), 36 pp. Available from Office of Technical Services, Washington, D.C.

Sodium chromate can be used effectively as a corrosion inhibitor for aluminum systems in concentrations as low as 0.1 ppm. The mechanism by which the chromate protects aluminum was also investigated.—NSA. 15174

**5.8.3, 6.2.2**

**Electrochemical Mechanism of the Inhibiting Action of Lead Oxides on Iron.** (In German.) J. D'ANS, W. BRECKHEIMER AND H. J. SCHUSTER. *Werkstoffe u. Korrosion*, 8, 677-688 (1957) November.

Lead dioxide is ineffective because its high electric conductivity causes local cells. Reaction compounds of divalent lead generate a protective coating on the basis metal which protects against corrosion. 7 references.—MR. 15236

**5.8.3, 5.9.2**

**Mechanism of the Inhibitor Action of Heterocyclic Nitrogen Bases in the Pickling of Steel in Sulfuric Acid in the Presence of Some Anions.** N. D. RUS'-YANOVA AND M. V. GOFTMAN. *J. Applied Chem., USSR* (Zhurnal Prikladnoi Khimii), 30, No. 10, 1564-1569 (1957). Available from Associated Technical Services, Inc., P. O. Box 271, East Orange, New Jersey.

Inhibitor action was shown markedly increased by presence of SCN<sup>-</sup>, I<sup>-</sup>, and Br<sup>-</sup> ions. 4 tables, 1 figure, 3 references.—ATS. 15422

**5.8.3**

**Studies on Amine-Type Corrosion Inhibitors (10th Report).** Behaviors of Inhibitors in Aqueous Solution. (In Japanese.) S. FUJII AND K. ARAMAKI. *Corrosion Engineering*, 6, No. 4, 205-209 (1957) July.

We have studied how alkyl and acyl groups of the inhibitor-molecules function as hydrophobic ones. In primary-amine type or in secondary-amine type inhibitors, the best inhibition in aqueous media are gained when they have four organic groups in total, attached to amines or acids in either cases.—JSPS.

**5.8.3**

**Studies on Amine-Type Corrosion Inhibitors (11th Report).** Filming Effects of Hydrocarbon Radicals. (In Jap-

anese.) S. FUJII AND K. ARAMAKI. *Corrosion Engineering*, 6, No. 6, 336-340 (1957) November.

Corrosion inhibitors consist of two functional groups. The one is atomic group functioning as adsorbent, another is organic radicals extending to metal surface which resist corrosives. Metals are protected from the penetration of corrosive materials by these barriers which are built by the organic radicals. So, the longer the organic radicals, the better inhibition can be obtained. When Oleoyl radicals are contained in the inhibitor molecule, some corrosive materials are included in the closed spaces which are formed by the oleoyl radical and the metal surface. Although corrosion proceeds rapidly in closed spaces, when the corrosives enclosed are exhausted, the reaction ceases. This means that corrosive materials from exterior spaces can be shut out. The same phenomena can be expected when the two organic radicals of C<sub>8</sub> inhibitor molecules are not connected to each other. However, C<sub>8</sub> inhibitor molecules did not give the same results. So, it is concluded that there are spaces through which oxygen, as chief corrosive in saline water, can penetrate. Diamine-type inhibitors cannot be adsorbed on metal surfaces in spite of two adsorbent radicals.—JSPS. 15258

## 6. MATERIALS OF CONSTRUCTION

### 6.3 Non-ferrous Metals and Alloys—Heavy

**6.3.20, 3.7.2, 3.7.3**

**Preliminary Report on Corrosion of Low-Uranium, Zirconium-Base Alloys.** A. E. DWIGHT AND A. H. ROEBUCK. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-5196, Oct., 1953 (Declassified Feb. 12, 1957), 31 pp. Available from Office of Technical Services, Washington, D. C.

Tests were made to determine the effects of heat treatment and composition on the corrosion resistance of low-uranium, zirconium-base alloys to water at 600 F. A total of 57 compositions were tested. The zirconium alloys contained up to 9% natural uranium plus small amounts of tin, antimony, lead, bismuth, yttrium, beryllium, germanium, niobium, nickel and aluminum in various combinations. Data are presented in both tabular and graphical form. The effect of heat treatment on corrosion resistance of zirconium-uranium-tin alloys is partially masked by impurities in alloys made from sponge or bomb-reduced zirconium. In ternary alloys made from crystal bar zirconium, the effect of heat treatment on corrosion resistance is definite, and varies with the composition of the alloy. The range of ternary compositions from approximately 2 to 6 wt% uranium and from 2 to 4% tin, are the most attractive fuel element core alloys. (auth.)—NSA. 14943

**6.3.20, 6.2.5, 4.3.3**

**Corrosion Resistance of Zirconium Metal and Stainless Steels on Exposure to Concentrated Solutions of Uranyl Fluoride.** C. D. SUSANO. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., Y-714, January 22, 1951 (Declassified March 6, 1957), 22 pp. Available from Office of Technical Services, Washington, D. C.

The results of exploratory tests to determine the degree of corrosion resist-



ance of zirconium metal and of various grades of stainless steels when exposed to highly concentrated solution of uranyl fluoride up to 100 C are reported. It is concluded that zirconium metal is not sufficiently resistant to merit consideration for application in homogeneous reactors. Some tests were made to determine the suitability of chromates, hydrogen peroxide and triethanolamine for use as corrosion inhibitors but with no apparent success. It is shown that under the particular conditions used all the various grades of stainless steels are equally and satisfactorily resistant to the corrosive attack of uranyl fluoride solutions at 100 C.—NSA. 15072

#### 6.3.20, 6.3.13

**Zirconium and Tantalum.** G. L. MILLER. *Chem. & Process Eng.*, **38**, No. 10, 389-390 (1957) October.

Tantalum was employed for special applications in chemical industry for many years and despite its cost and development of competitive materials, its use has expanded. Zirconium has made its name in nuclear industry, where combination of low thermal neutron capture cross-section and excellent corrosion resistance resulted in its employment as a canning material in packaged power type of reactor. Both metals have excellent corrosion resistance in most acids, but tantalum is attacked by alkalis while zirconium is not. Corrosion data for zirconium in hydrochloric acid show penetration figures less than 0.0002-in. per year for all concentrations up to boiling point with 20% acid. Up to 25% acid at 325 F, resistance is excellent. At 400 F 15% concentration results in a corrosion rate of less than 0.005-in. per year. Tantalum will not be easily and quickly supplanted as a material for specially severe corrosive conditions, nor stainless steel as a relatively cheap material usable for less severe conditions. Tables.—INCO. 15013

#### 6.3.20

**Some Properties of Zirconium-Niobium Alloys.** YU. F. BICHKOV, A. N. ROZANOV AND D. M. SKOROV. *J. Nuclear Energy*, **5**, 402-407 (1957).

The constitution of zirconium-niobium alloys has been investigated and modifications to the existing equilibrium diagram are suggested. The mechanical properties of the alloys at room temperature have been determined and the hot-strength at temperatures of up to 750 C has been deduced from hardness measurements. Also, hardness has been measured after low-temperature ageing treatments. Measured results are correlated with the equilibrium diagram. The resistance of the alloys to oxidation in air has been determined in the temperature range 570 to 650 C. (auth.)—NSA. 15204

### 6.4 Non-ferrous Metals and Alloys—Light

#### 6.4.2

**Study of Aluminum Alloys Containing up to 45 w/o Uranium.** NORMAN E. DANIEL, ELLIS L. FOSTER, JOHN A. DEMASTRY, ARTHUR A. BAUER AND RONALD F. DICKERSON. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-1183, April 30, 1957 (Declassified June 4, 1957), 40 pp. Available from Office of Technical Services, Washington, D. C.

Casting techniques for the production of sound homogeneous extrusion blanks composed of aluminum-uranium alloys

with 30 to 45 wt% uranium were investigated. Satisfactory extrusion blanks in the form of sleeve castings containing 30 wt% uranium were produced utilizing air-melting techniques. Sound casting 2 in. in diameter containing 45 wt% uranium were produced by conventional vacuum-melting techniques utilizing zirconia crucibles. An extrusion blank containing 45 wt% uranium with side walls 0.55-in. thick was vacuum melted and cast. This casting was essentially sound, porosity being found only in the area of the pour and was attributed to problems in mold design. As a supplement to the casting program, exploratory investigations were made of the fabricability and the corrosion resistance of the alloys. The diffusion rate of the uranium from the alloys into 2S aluminum cladding was also studied. All of the alloys, enclosed in heavy-walled-aluminum tubes, were pressed forged at 800 and 1000 F. The corrosion resistance of the high-uranium alloys is comparable to that exhibited by the aluminum-16 wt% uranium alloy and diffusion of the uranium from the alloys into 2S aluminum after 30 days at 750 F could not be detected by metallographic techniques. The effects of a 0.5 wt% silicon addition on the melting characteristics of the aluminum-16 wt% uranium alloy were negligible when compared with the effects of small variations in the uranium content. (auth.)—NSA. 14922

#### 6.4.2

**Corrosion-Resistivity of Anti-Corrosive Aluminum and Its Alloys.** (In Japanese.) HIROSHI NAKATSUGAWA. *Furukawa Electric Review*, No. 16, 33-48 (1957) Nov.

In view of the fact that aluminum and its alloys are in increasingly extensive use for architectural and other purposes, their corrosion resistance poses a most important item for research. This report covers a comparative study of the anti-corrosiveness of eight kinds of metals, such as high-purity Al, 1S, 2S, 3S, 61S, 52S, 56S and NP 5/6 and the relationship between their fabricating process and corrosion resistance and also between the period of their corrosion and the rate at which it progresses. The report clarifies the following matters. (1) We cannot positively conclude that the corrosion-resistance decreases in proportion to the decrease in the degree of workability. In fact, H18 and 0 (soft or annealed) have the same degree of corrosion resistance, while H12, H14, and H16 tend to be inferior in this respect. (2) During the 24-week period of corrosion, the metals developed noteworthy corrosion in the first 6 weeks, but, thereafter, their corrosion did not progress at any noticeable rate. (3) For all the aluminum alloys referred to, no considerable difference is recognizable in corrosion resistance viewed from the angle of surface contamination, or decrease in tensile strength and elongation. (auth.)—ALL. 15395

#### 6.4.2

**New High-Strength Aluminum Alloy.** R. ZUECH AND R. G. CRON. *Light Metal Age*, **15**, No. 1/2, 23, 26 (1957).

Physical and mechanical properties of 42B, a high-strength aluminum casting alloy developed by Zuech and Cron for North American Aviation Inc. are tabulated and discussed. Composition is not given, but the chief alloying elements are stated to be silicon and magnesium, with small additions of beryllium and sodium. Min. UTS of 42,000 and 45,000

lb./in.<sup>2</sup>, respectively are claimed for sand-cast and permanent-mould-cast specimens and ductility, corrosion-resistance and dimensional stability are good. Owing to the beryllium addition, the cost of 42B is moderately high compared with other aluminum alloys but the material is economical in practice, as it can replace relatively expensive forgings. A guide to casting of the alloy is presented.—MA. 15219

### 6.6 Non-metallic Materials

#### 6.6.8, 7.4.1

**Plastic Heat Exchangers Gain in Severe Service.** D. M. WHITLEY. *Haveg Industries. Chem. Eng.*, **64**, No. 9, 308, 310, 312, 314, 316 (1957) Sept.

Corrosion resistance, flexibility of design and low cost are advantages claimed for plastic heat exchangers. Plastic units compete successfully with least expensive stainless steel heat exchangers and for some time have even been less costly than copper, Monel or nickel. Workhorse plastics—phenolics and furans, carbon or asbestos filled—cover majority of applications, particularly for tube sheets and shells. Phenolics have safe range of use to 300 F. Glass reinforced polyester is used where higher pressures are encountered. PVC lined steel has strength but the PVC limits it to continuous service at 212 F and intermittent service at 265 F. For tubes, impervious graphite is recommended to 338 F. Heat transfer characteristics are discussed and typical design condition followed through, showing effects of film resistance and thermal conductivity in liquid-liquid, gas-liquid exchange situations for steel, stainless, brass, impervious graphite and glass materials. Cost curves for simple standard units are shown.—INCO. 15149

#### 6.6.8

**Build or Repair with Epoxy-Glass Laminates.** T. G. NOCK AND R. A. CODERRE. *Shell Chemical Corp. Chem. Eng.*, **65**, No. 2, 148, 150, 152 (1958) Jan. 27.

Epoxy laminates are now used in fabrication of equipment for chemical service such as tanks, pipe, fume hoods, ducts and exhaust stacks. In unusual new technique, basic materials—epoxy resins and glass cloth—can be used for preparing durable patches for process metal pipe, tanks and pressure vessels. Important properties and applications are reviewed.—INCO. 15164

### 6.7 Duplex Materials

#### 6.7.2

**Alumina-Base Cermets, Part 4.** J. W. LINDENTHALL, J. G. STRADLEY AND T. S. SHEVLIN. The Ohio State University Research Foundation. U. S. Wright Air Development Center, May, 1957, 19 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C.

Strong, nonporous, well-bonded alumina-base cermets containing 50 volume percent of oxide have been developed during Air Force-sponsored studies of the wettability of aluminum oxide by various metals. A report of the development has just been released to industry through the Office of Technical Services, U. S. Department of Commerce.

The studies were made by pre-reacting alumina with easily reducible oxides such as those of chromium, nickel and cobalt. Compounded into cermet compositions, these mixed oxides promoted

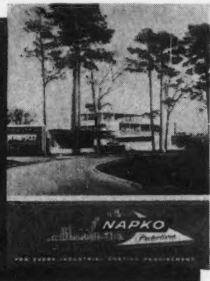


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wetting or reaction with the metallic constituents.

Specimens containing 50 volume % of iron and 50 volume % of oxide were developed. They were nearly nonporous, as strong as the metal alone, and their high elastic moduli indicated good bonding. It was suggested that compositions containing more than 50% by volume of metal could be produced.

Cernets composed of metals & alumina with chromic oxide were fired successfully in a hydrogen atmosphere. In the case of alumina and nickel oxide, it appeared that nonporous specimens might be prepared in an atmosphere other than hydrogen or vacuum, possibly helium.—OTS. 15273

#### 6.7.2, 3.2.2

Observations on the Brittle Fracture of Cemented Titanium Carbide. J. R. Low, Jr. *J. Metals* (Trans. AIME), 8, No. 8, Sec. 2, 982-984 (1956) August.—BNF. 13266

### 7. EQUIPMENT

#### 7.4 Heat Exchangers

##### 7.4.1, 6.4.2

Aluminium Alloy Variety Spurs Use in Exchangers. R. W. FLOURNOY. *Chemical Engineering*, 64, No. 10, 318-320, 322, 324 (1957) October.

Those mechanical and chemical properties of a variety of aluminum alloys which justify their growing acceptance as a construction material for heat exchangers are reviewed. The cost comparison of such aluminum heat exchangers with those made of other metals is tabulated. Fabrication and design techniques, with special emphasis on the necessity of avoiding bimetallic couples are discussed. Aluminum heat exchangers and condensers are recommended for use in the following types of corrosion environments: Sea water, fresh water, distilled and demineralized water; petroleum products; liquid oxygen, peroxide solutions, nitric acid; organic solvents such as benzene, toluene, alcohol, ether and acetone; fatty acids and oils; food products and pharmaceuticals; ammonia, ammonium nitrate and ammoniated ammonium nitrate; carbon dioxide and carbonic acid solutions; sulfur, mercaptans, hydrogen sulfide and hydrogen cyanide.—ALL. 14900

##### 7.4.1

Cooling-Tower Water Treatment. M. J. GOSSOM AND J. O. JOHNSON. *D-X Sunray Oil Co. Oil & Gas J.*, 55, No. 49, 91-95 (1957) Dec. 9.

Almost exclusive use of shell and tube equipment in modern cooling water systems emphasizes need for clean heat-transfer surfaces. Modern chemical treating procedures for prevention of scale, corrosion, slime and algae and other associated waterside problems require intelligent supervision for good results. Several treatments provide excellent corrosion and scale prevention opportunities at a reasonable cost provided certain conditions can be met. Maintenance of circulating water and acid feeder at D-X Sunray Oil Co. is described. Photos, graphs, diagrams.—INCO. 15292

#### 7.7 Electrical, Telephone and Radio

##### 7.7, 8.4.3, 5.4.5

How to Combat Corrosion of Equipment. R. S. BIRMINGHAM. Paper before Natural Gasoline Assoc. of America, Rocky Mountain Mtg., 1957. *Oil & Gas J.*, 56, No. 2, 113 (1958) Jan. 13.

Atmospheric conditions existing in areas of sour-gas and oil production and processing usually play havoc with exposed electrical equipment and copper wiring. Standard mercury meters have been found satisfactory in sour gas measurement. Corrosion does take place in meter-pressure shaft bearings unless plastic-sealed bearings, such as Teflon, are used. Pneumatic transmitters having Type 316 stainless measuring diaphragms proved to be very satisfactory for sour-gas measurement and control purposes. All meters, transmitters and controller assemblies that are subject to corrosion are protected by painting with Duco lacquer. Type 316 has proved very satisfactory for all sour-service control valve trim, meter and controller elements. Type 304 was found to be generally unsatisfactory and its use was soon discontinued.—INCO 15178

### 8. INDUSTRIES

#### 8.3 Group 3

##### 8.3.1, 5.4.5

Rusting and Its Cost to Farming. E. E. WHITE. *Corrosion Technology*, 4, No. 12, 413-416 (1957) Dec.

Reviews types of steel used for farm buildings, or parts of buildings and for mobile or stationary machinery; forms of protective measures applicable (paints, metallic aluminum and zinc sprays, galvanizing); and proper surface preparation. Efficient painting system (which should last 7-10 years) should consist of 2 coats of priming paint, one of undercoat and one finishing coat, producing thickness of at least 0.005-in. Photographs show fertilizer discs with various anti-corrosion treatments before and after use.—INCO. 15140

##### 8.3.5

Corrosion and Food Manufacture. J. W. SELBY. *Corrosion Prevention & Control*, 4, No. 9, 37-40, 46 (1957) Sept.

Background for discussion of corrosion problems in food industry is provided by consideration of changes in flavor, odor and appearance and possibility of toxicity, due to metallic contaminants in food. Discussion covers: natural inhibitors in foods (fat, gelatin, sugar, sodium nitrate formed in cured meats); use of stainless steel equipment; use of copper in jam-making industry (where sulfur dioxide is used as preservative) and in manufacture of boiled sugar confectionary; problems encountered and materials used in handling meat products and cured meats; corrosion of tinplate cans and use of lacquers and dip-treatments; and use of plastics.—INCO. 15082

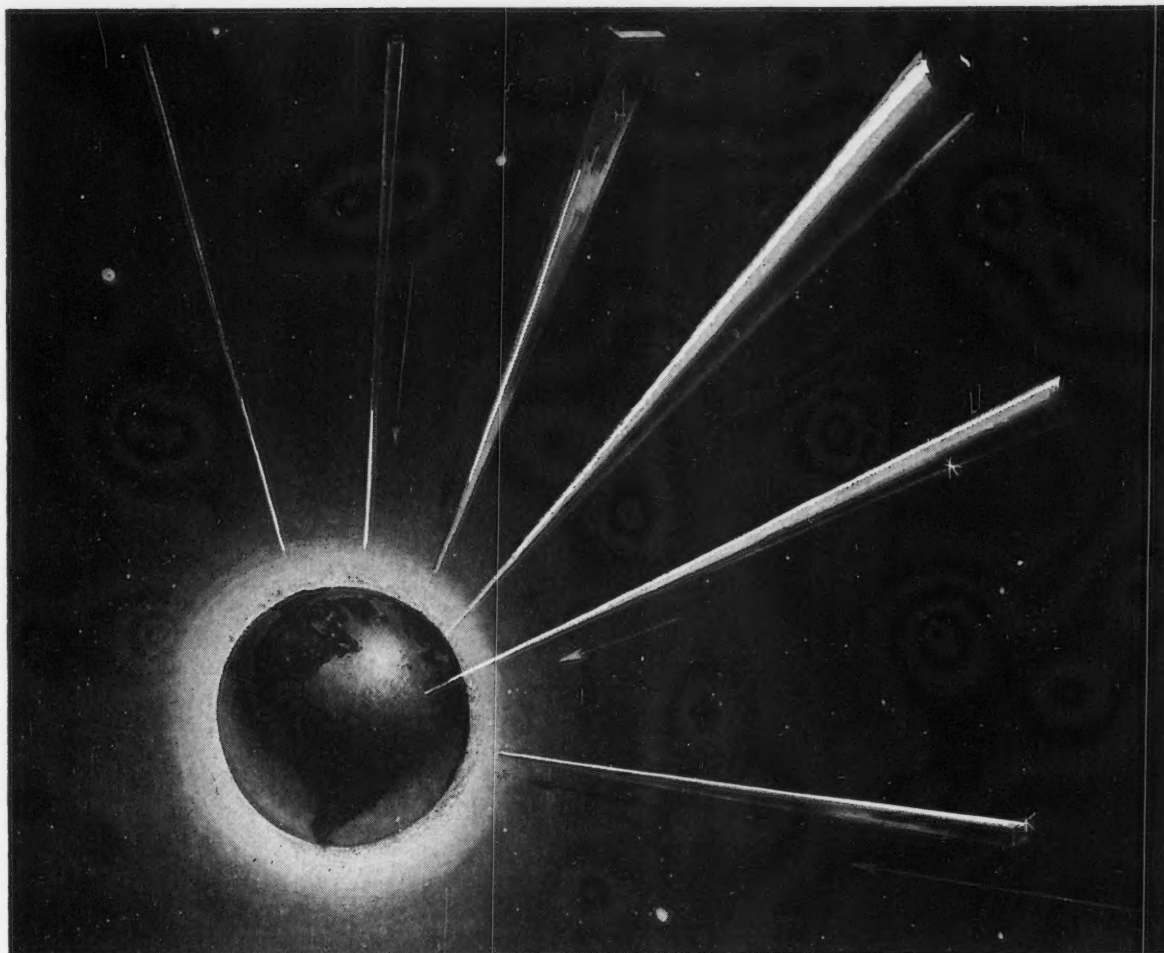
#### 8.4 Group 4

##### 8.4.5, 3.5.4, 6.2.5

Radiation Damage Experiments for the Homogeneous Reactor. C. H. SECOR. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-51-4-57, April 13, 1951 (Declassified Feb. 14, 1957), 11 pp. Available from Office of Technical Services, Washington, D. C.

Tests of the effects of irradiation upon uranyl sulfate solutions were conducted using a highly enriched uranyl sulfate solution in light water contained in a Type 347 stainless steel system. The solution is similar to that proposed as a fuel and heat transfer medium for the Homogeneous Reactor Experiment. Re-





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sults of stability tests on the solution during irradiation are presented. Extensive studies of the corrosive effect of the solution showed excellent corrosion resistance of 18-8 stainless steels and of titanium at 100 C, but at 250 C corrosion of stainless steel accompanied by reduction of the uranyl ion resulted. Tests showed that pretreatment of the steel with either nitric or chromic acid provided a protective film of hydrous oxides, and prevented reduction of uranyl ion and loss of uranium from the solution after 10 months' trial. Although such a protective film appeared adequate for the operation of a homogeneous reactor, 22 in-pile experiments were conducted to study the effects of radiation on the corrosion of the steel container and on the stability of the solution. Conditions for pretreatment for each of the 22 experiments, approximate temperature during irradiation and duration of irradiation are given in table form. Results show that the corrosion behavior

and solution stability is improved by the presence of pile irradiation. A possible explanation of this behavior is offered. Final tests of the behavior of circulating systems will be made in the Homogeneous Reactor Experiment.—NSA. 15113

#### 8.4.5, 3.7.2, 6.4.2

**Boron—Aluminum and Boron—Uranium—Aluminum Alloys for Reactor Application.** W. C. THURBER, J. A. MILKO AND R. J. BEAVER. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-2149, October 17, 1957, 25 pp. Available from Office of Technical Services, Washington, D. C.

Additions of small quantities of the burnable poison, boron, in aluminum reactor fuel elements offer the possibility of reducing undesirable neutron-flux perturbations, especially those caused by fuel burnup. This report describes techniques for induction melting of boron-aluminum and boron-uranium-

aluminum alloys for producing materials with maximum homogeneity. Of the several boron additions investigated, a nominal 12 wt % boron-nickel master alloy was the most satisfactory for preparing boron-uranium-aluminum castings, while both 1.5 to 5 wt % boron-aluminum and 12 wt % boron-nickel master alloys were suitable for preparing boron-aluminum castings. Data are presented which indicate that boron improves the strength of aluminum. The corrosion resistance of a nominal 0.1 wt % boron-0.8 wt % nickel-aluminum alloy is comparable with type 1100 aluminum in distilled and aerated water at both 60 and 100 C. Without the nickel, this alloy appears to be marginal in these environments. (auth)—NSA. 15123

#### 8.4.5, 4.3.2, 6.3.21


**The Dissolution of Thorium Metal and Thorium Dioxide in Nitric Acid-**

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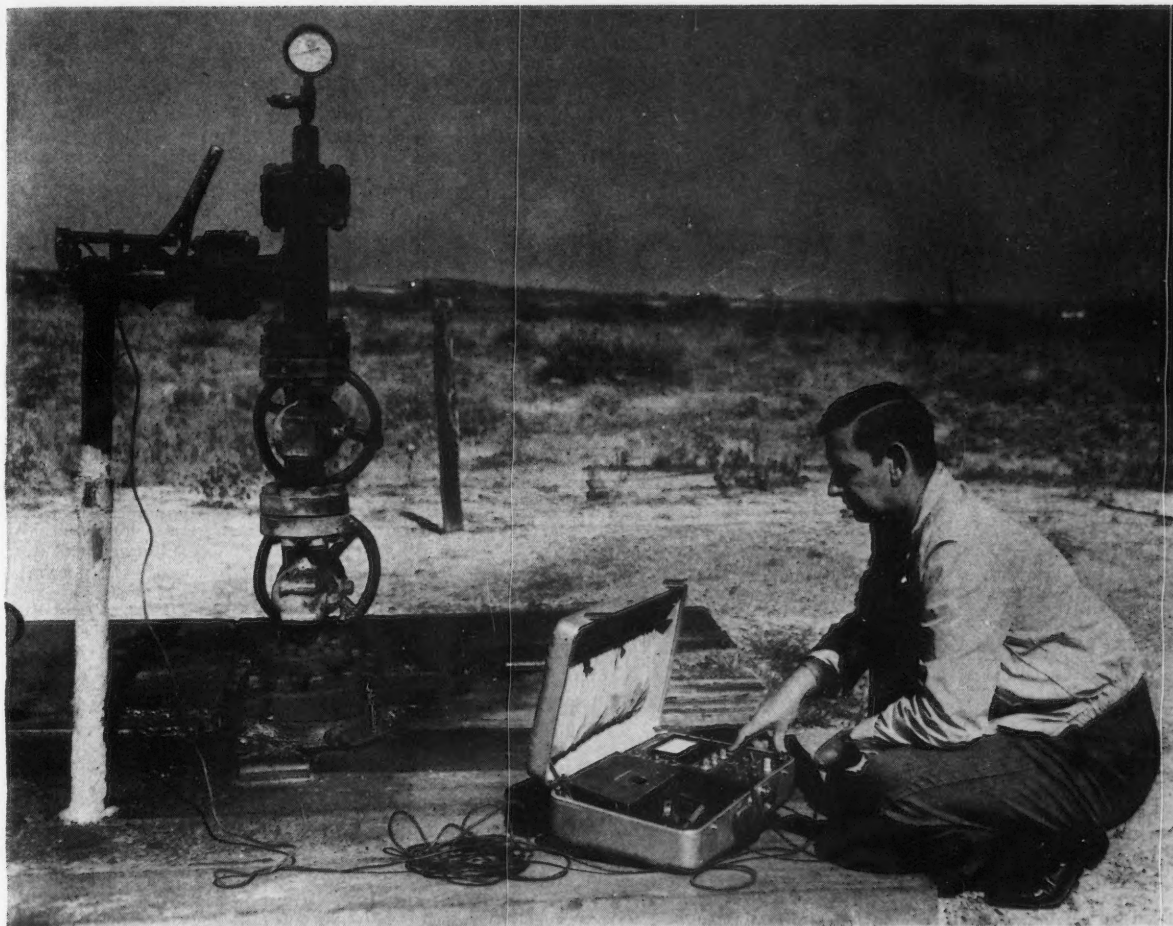
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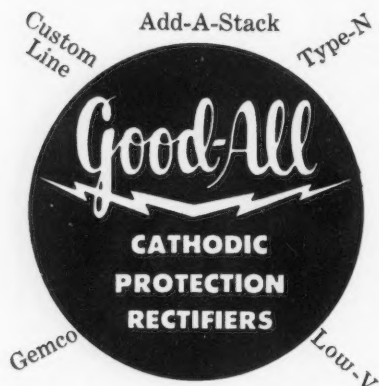
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**hydrofluoric Acid and Nitric Acid-Ammonium Fluosilicate Mixtures.** Work Done September 15, 1944-June 30, 1945. F. W. SCHULER, F. L. STEAHLY AND R. W. SROUGHTON. Chicago University, Metallurgical Lab. U. S. Atomic Energy Commission Pubn., CC-3576, August 2, 1946 (Declassified Feb. 15, 1957), 16

pp. Available from Office of Technical Services, Washington, D. C.

A satisfactory method has been found for dissolving thorium metal and thorium dioxide in 25-12 heat-treated stainless steel vessels without excessive corrosion of the vessels. The dissolution medium consists of strong nitric acid containing a small amount of fluoride or fluosilicate. The fluoride required has no harmful effects on a subsequent solvent extraction step for a separation of uranium isotopes from the dissolved thorium. (auth.)—NSA. 15401

stabilized, have been tested in synthetic filtrates and found to corrode at the rates 0.007 and 0.045 mil/year, respectively. These values are much smaller than an estimated maximum allowable corrosion rate of 10 mil/year. It is concluded that either of these stainless steels or 304 or 316 stainless steels could be used in the construction of tanks for storing acid filtrates from the UAP process. (auth.)—NSA. 14927



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#### 8.4.5, 4.3.3

**HRT CP Experimental Evaporator Studies—Problem Statement.** J. C. BRESEE. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., Cf-55-2-82, Feb. 11, 1955 (Declassified Feb. 14, 1957), 12 pp. Available from Office of Technical Services, Washington, D. C.

To recover the deuterium oxide from the solution removed from the HRT core, it is proposed that the solution be evaporated to approximately 50 wt% uranyl sulfate in the HRT chemical plant, thereby effecting 97% recovery of the deuterium oxide. A study is made of the evaporation from 1.6 to 50 wt% uranyl sulfate including problems of entrainment and condensate contamination, evaporator control, solution removal and corrosion. Designs for experimental evaporators are included.—NSA. 14957

#### 8.4.5, 4.3.3

**Corrosion of Stainless Steels in Uranyl Ammonium Phosphate Filtrates from Hanford Waste.** H. A. BERNHARDT, W. DAVIS, JR., AND J. R. FLANARY. Carbide and Carbon Chemicals Corp. U. S. Atomic Energy Commission Pubn., K-405, June 1, 1949 (Declassified Feb. 23, 1957), 13 pp. Available from Office of Technical Services, Washington, D. C.

Storage of acid filtrates from the UAP process for uranium recovery from alkaline Hanford waste has imposed the problem of determining the type of metal that can be used for storage tanks. Two stainless steels, 302 and niobium

#### 8.4.5, 4.7

**Liquid Metal Fuel Reactor Fuel Processing Studies.** C. RASEMAN, R. BAUMAN, AND J. WEISMAN, Compilers. Brookhaven National Lab. U. S. Atomic Energy Commission Pubn., BNL-345, January 1955 (Declassified March 18, 1957), 22 pp. Available from Office of Technical Services, Washington, D. C.

Small scale studies and loop tests were carried out on bismuth-cerium-uranium/lithium chloride-potassium chloride systems to estimate the effect of uranium and cerium concentration on the respective distribution coefficients, the effect of metal-metal ion additives, the effect of redox additives and the distribution coefficients of the nonvolatile fission products obtained by pile irradiation. The corrosion of stainless steel by liquid bismuth-uranium alloys was investigated.—NSA. 15402

#### 8.4.5, 4.7

**Progress Report for May 1951. North American Aviation, Inc.** U. S. Atomic Energy Commission Pubn., NAA-SR-Memo-56 (Del.), July 15, 1951 (Declassified with Deletions Feb. 27, 1957), 23 pp. Available from Office of Technical Services, Washington, D. C.

Reactor calculations, materials and liquid metal system corrosion for an optimized plutonium power reactor are briefly summarized. Results of a heat transfer study for a high temperature reactor are tabulated. Preliminary results on the corrosion of molybdenum, tantalum and graphite in liquid metals are also tabulated. Work was continued on the mechanical properties of materials at high temperatures. Some properties of graphites are listed. Fuel preparation techniques for the Low Power Research are summarized. Exponential experiments are described. Data are included on the effect of cold work and irradiation on the low temperature electrical resistivity of copper. Surface hardness measurements on 347 stainless steel welds in NaK are summarized. Work on the Materials Testing Accelerator was also continued.—NSA. 15021

#### 8.4.5, 5.7.7, 5.9.4, 3.2.2

**Shielding Reactor Corrosion Studies.** ARNOLD R. OLSEN. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-967, July 9, 1951 (Declassified March 2, 1957) 10 pp. Available from Office of Technical Services, Washington, D. C.

The investigation of corrosion in filtered water of MTR-type fuel elements simulated in this study by samples of 2S and 72S-clad 2S aluminum and an aluminum brazing alloy for the Shielding Reactor is reported. The study included two basic types of protection Element pretreatment, by either anodizing or alodizing; and solution control, using nitric acid to maintain a pH of 5.5 to 6.5 or the addition of 60 ppm of sodium chromate or potassium chromate as an inhibitor. No pretreatment was effective in stopping pitting attack for more than four months as a maximum. Control of

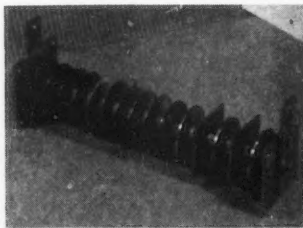
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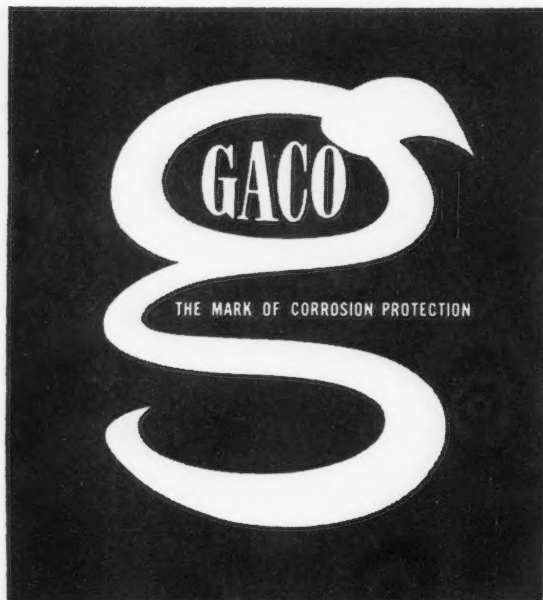
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the pH tended to reduce pitting attack and at the same time reduce the over-all corrosion rate to essentially zero. (auth).—NSA. 15006

#### 8.4.5, 5.8.2

**Decontamination of HRE Loop.** Quarterly Report for Period November 1952 to January 1953. R. J. McNAMEE. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-53-2-75, Feb. 6, 1953 (Declassified Feb. 16, 1957), 14 pp. Available from Office of Technical Services, Washington, D. C.

The corrosion characteristics of 347 stainless steel piping for the HRE were investigated. Specifically, several solutions and corrosion inhibitors were tested in order to develop a satisfactory procedure for removing radioactive fission product contaminants. Two solutions were found which appeared satisfactory: Scale-Loosening Solution No. 2 (1.2M hydrochloric acid and 1.8M sulfuric acid), and Pickling Solution No. 15 (1.2M hydrochloric acid and 0.7M Phosphoric acid). Alkyl Pyridines HB was shown to be a satisfactory corrosion inhibitor for each solution. Solution No. 15, inhibited with Alkyl Pyridines HB, refluxed at 80 to 85 C, gave a corrosion rate of 33 mils/yr.—NSA. 14971

#### 8.4.5, 6.2.5

**Reactor Materials.** Chapter 10. G. E. EVANS. Oak Ridge School of Reactor Technology. U. S. Atomic Energy Commission Pubn., CF-52-8-148 (Chap. 10), December 23, 1952 (Declassified Feb. 16, 1957), 45 pp. Available from Office of Technical Services, Washington, D. C.

Stainless steels and their compositions and properties are described and tabulated. Corrosion behavior, impact strength, stress rupture, tensile strength, yield strength, creep, hardness and elasticity are some of the properties included.—NSA. 14936

#### 8.4.5, 6.2.5, 4.6.2

**The Corrosion Behavior of Various Stainless Steel Boron Alloys in 680 F. Water for Use as a Possible Burnable Poison Element.** DONALD C. BEELQUIN. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-M-DCB-2, May 28, 1957, 13 pp. Available from Office of Technical Services, Washington, D. C.

Burnable poison specimens of stainless steel containing up to 0.96 wt % boron were exposed to high purity water at 680 F (2700 psi). The results indicate that neither the fabrication methods used or boron additions within the range investigated alter the corrosion resistance of unalloyed stainless steel. (auth).—NSA. 14934

#### 8.4.5, 6.3.17

**Resume of Uranium Alloy Data-X.** E. F. LOSCO AND Z. M. SHAPIRO. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-PWR-PMM-282, Nov. 14, 1955 (Declassified March 7, 1957), 67 pp. Available from Office of Technical Services, Washington, D. C.

The principal developments in the PWR uranium fuel program consisted of the bond-line attack in coextruded lithium-uranium system, aluminum-base alloys as barrier layers for U<sub>3</sub>Si fuel elements, corrosion mechanism for bare silicon-uranium systems, irradiation, induced transformation in molybdenum-

uranium and niobium-uranium alloys  
uranium dioxide irradiation program.—  
NSA. 14983

#### 8.4.5, 6.3.20

**Effects of Radiation on the Electrochemical Activity of Zirconium.** (In Russian.) I. L. ROSENFELD AND E. K. OSHE. Inst. of Physical Chemistry Acad. of Science U.S.S.R. *Doklady Akad. Nauk SSSR*, 114, 143-145 (1957) May 1.

The relationship between electron radiation intensity and electrochemical activity was evaluated for zirconium-aluminum and zirconium-iron couples in 3% sodium chloride.—NSA. 15044

#### 8.4.5, 7.2, 6.2.5

**Piping Systems and Metallurgy for Nuclear Applications.** *Power Eng.*, 61, No. 12, 114, 116, 118 (1957) Dec.

Discussion of basic mechanism of radiation damage and how it affects piping design and metals used in piping systems. Except in primary loops handling highly radioactive fluids much of piping in a nuclear power plant does not differ from that in a conventional steam plant. At Argonne nuclear plant extensive use was made of stainless steel to reduce number of corrosion and erosion particles. Piping, valves and fittings of auxiliary systems are of stainless steel but because of high cost of this material, design provided that only piping in feed water and steam systems connected directly to reactor be of the material. Thermal insulation of the more highly radioactive parts of these piping systems is of stainless steel wool since normal insulating material would break down under radiation. For each type of reactor primary loop piping presents a different corrosion problem. In case of water cooled reactors radiation provides a constant source of dissolved oxygen which together with decomposition products are factors in steel corrosion. Since reactor piping in general must provide for long operation, accumulation of corrosion products carried from piping to reactor is intolerable and thus pipe and valves must be of corrosion resistant materials. In case of liquid sodium heat transfer, there also exists a problem of attack upon steel and nickel-chromium austenitic steels are deemed necessary to resist it. Where liquid bismuth is the medium, there are mass transfer considerations and chromium-molybdenum ferritic steels have given most favorable performance characteristics to date. Perhaps most extreme piping corrosion is that to be contended with in aqueous homogeneous systems, where highly resistant lining materials are sought. Photos.—INCO. 15050

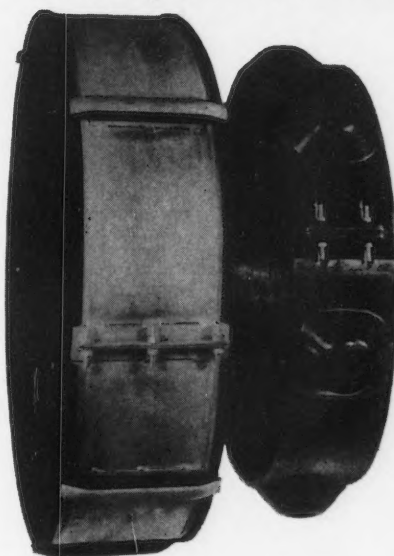
### 8.8 Group 8

#### 8.8.1

**Large-Scale Continuous Production of Ammonium Perchlorate.** J. C. SCHUMACHER AND D. R. STERN. American Potash and Chem. Corp. *Chem. Eng. Progress*, 53, No. 9, 428-432 (1957) Sept.

Describes an existing large-scale modern plant for production of ammonium perchlorate, including details of process, instrumentation and operating variables. Preliminary experimental work on solubility, safety considerations and materials of construction is discussed. All welded piping and medium size vessels are of heat-treated 316 stainless. Sections of plant in contact with excess hydrochloric acid were constructed of glass lined steel or Hastelloy C. Table

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gives corrosion rates in ammonium perchlorate at 80 C of Hastelloys, 300 series stainless steels, Inconel, nickel, Monel and cast iron. Photos, diagrams, graphs. —INCO. 15419

#### 8.8.5, 3.7.3

**Defects in Plated Zinc-Base Die-Castings.** F. RICHTER. *Metall.*, 11, No. 6, 480-484 (1957); *Bull. Brit. Non-Ferrous Metals Research Assoc.*, 37, No. 338, 352 (1957).

Various defects, arising either in the casting or the plating process are discussed in the light of photo-micrographs. The article then deals with the effect of heating during the lacquering process on the adhesion of the plated coatings. —RPI. 15048

#### 8.8.5

**Composite Castings of Aluminium-Bronze and Steel: Improved Corrosion-Resistance and Stiffness.** C. H. MEIGH AND G. E. E. MARSHALL. *Engineering*, 183, No. 4757, 594-595 (1957).

The development of composite castings made by casting aluminum-bronze on to a steel core enables the corrosion-resistance and non-sparking characteristics of aluminum-bronze to be combined with the low cost and high strength of steel. The design and production of such castings are described, and the application of the technique to the manufacture of pneumatic diggers and rotary pumps is discussed.—MA. 14996

### 8.9 Group 9

#### 8.9.3, 5.4.5

**What are the Qualities of a Good Asphalt Pipe Coating.** LLOYD F. BRAM-

BLE. *Pipe Line News*, 29, 63+4 pages (1957) Sept.

Considers the entire complex of deteriorative influences which come into play the moment a pipeline is cleaned, coated, wrapped, lowered into the ground, and backfilled.—BTR. 14954

#### 8.9.3, 5.2.3, 4.2.7

**Venezuela's Corrosion Problem Is a Special One.** C. R. LANDERS. Venezuelan Atlantic Refining Co. *Petroleum Engr.*, 29, No. 12, D28-D31 (1957) Nov.

Discussion of external corrosion control of oil and gas pipelines in Venezuela includes information of value in any area of similar soil and climatic condition. Pipelines are being operated in jungles and rain forest of eastern Venezuela, the llanos of north central area, Coastal Range along north coast line, in foothills of Andes and along coast and under waters of Lake Maracaibo. During rainy season rights-of-way are impassible and during dry season rate of corrosion is reduced to point that detection is almost impossible. Soil resistivity surveys conducted during dry seasons are worthless; if cathodic protection with rectifiers is contemplated, current requirement test conducted during rainy season will provide most valuable information available. Most widely employed corrosion mitigation device is line blocking (placing of supports under surface pipelines); various types of support designs are discussed. In cathodic protection, cast magnesium anodes are limited to protection of river and road crossings. At Lake Maracaibo, combination of aluminum pipe, concrete jacket and installation below mud-line combats problem of teredo.—INCO. 14981

#### 8.9.3, 5.4.5

**Internal Surface Coating of Pipe.** G. G. WILSON AND R. T. ELLINGTON. *Inst. Gas Technology. Gas*, 33, No. 7, 107-108, 112, 115-116, 121 (1957) July.

Major portion of pipeline dust is Fe oxide, formed by corrosion of pipe surface which can damage pipe fittings and regulator valves by abrasion, contribute to inaccurate orifice metering by adhering to or eroding edge of orifice plates and cause faulty operation of appliances through stoppage of orifices. Presence of water is essential to corrosion process and corrosion is promoted by presence of hydrogen sulfide, oxygen and carbon dioxide. Dehydration and desulfurization of natural gas at processing plants prior to its transmission proved effective in eliminating internal corrosion and in maintaining flow efficiency of gas transmission pipelines. Method of preventing interior corrosion of pipe by coating inside surface with a corrosion resistant material is discussed. Survey of literature and industry practice was made to determine present state of art of internally coating pipe and ascertain which types of plastic coating materials could be expected to withstand gas pipeline service conditions. Industrial experience, methods of applying coatings, materials recommended for interior coating and results of laboratory tests on coating materials are given. Photos, table. —INCO. 15153

#### 8.9.4, 6.2.5

**Alloy and Special Steels in Railway Work.** H. O'NEILL. Paper before Symposium on Alloy Steels, Nat. Metall. Lab., Jamshedpur, India, 1956. *Metalurgia*, 56, No. 334, 74-76 (1957) August.

Refers to applications of alloy steels to locomotives, motion parts, drawgear, springs, tires, rails and cars. Use of boiler shell and firebox containing 1.75-2.0 nickel enabled saving of weight of over 2 tons with service design based on tensile strength; when nickel is in short supply, good quality higher manganese steel would suffice. In motion parts, a nickel-chromium-molybdenum steel of 50-60 tsi ultimate tensile stress saved 1000 lb. weight compared with 40-45 tsi manganese-molybdenum steel. Chromium steel rail with structure of tempered martensite gave best wear on sharp curves. Austenitic 12 manganese steel rail gives excellent service at busy crossings. Chromium-nickel austenitic inserts (245 Brinell) welded from Armex 2 electrodes into running surfaces of ordinary rails gave remarkable wear resistance. Copperbearing steel rails corrode less in open air than plain steel rails; this does not apply to steam-traffic tunnels.—INCO. 15007

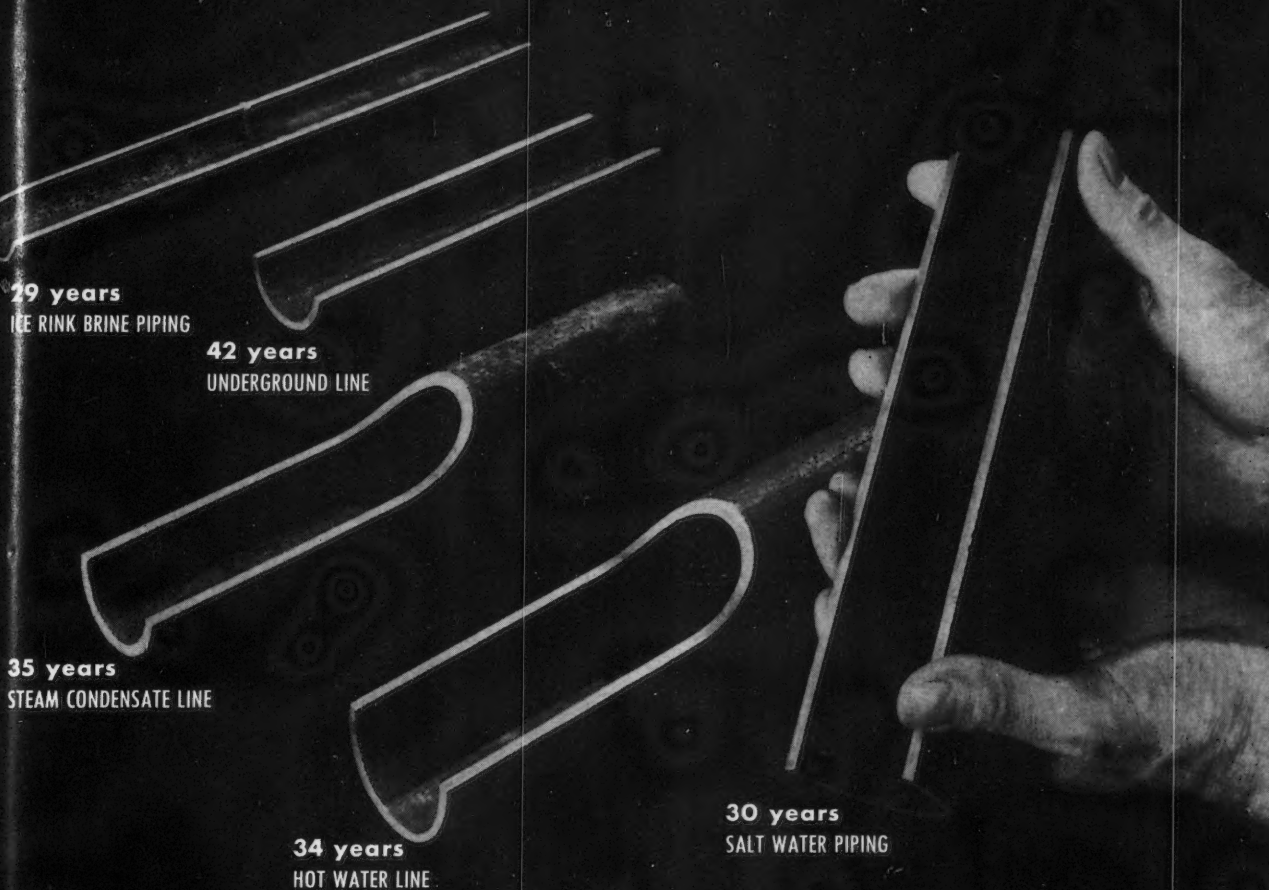
#### 8.9.4, 4.4.9

**Performance of Residual Fuels in High-Speed Diesel Engines.** D. R. JONES, K. L. KIPP AND J. E. GOODRICH. Paper before Am. Soc. for Testing Materials, Second Pacific Area National Meeting, Symposium on Railroad Materials, Los Angeles, September 18-19, 1956. ASTM Special Technical Pubn. No. 214, 51-60, disc., 61-62 (1957).

To investigate high wear obtained with residual fuels wear tests were carried out in single-cylinder laboratory engines equipped with radioactive piston rings. Data on effect of operating conditions on wear showed that increasing jacket temperature and engine load reduced wear. In residual fuels sulfur was



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found to have same absolute effect on wear as in distillate fuels. However, because wear was much higher with residual fuels, relative effect of sulfur was substantially less than with distillate fuels. Removal of abrasive contaminants in residual filtration or centrifuging significantly reduced wear. Viscosity by itself was found to have little or no effect on wear in these laboratory tests. Tables, photos.—INCO. 15323

## 8.9.4

**Operation of Diesel Locomotives with Dual-Fuel System.** P. V. GARIN. Paper before Am. Soc. for Testing Materials, 2nd Pacific Area Nat'l. Meeting, Symposium on Railroad Materials, Los Angeles, Sept. 18-19, 1956. ASTM Special Technical Pubn. No. 214, 3-46; disc. 47-50 (1957).

Results obtained from dual-fuel research project to date have indicated feasibility of utilizing two fuels on a locomotive Diesel engine, 1 fuel having necessary characteristics for good starting, idling and low output operation and other a less critical fuel which will provide effective high output operation. Spectrographic analyses of fuels X-3 and X-6 include nickel value. Cylinder liners used in both fuel control units were reclaimed by chromium plating in accordance with normal practice. Two types of plating were used, pocket-type chromium and channel-type chromium. Some of the former liners were built up with an underlay of iron plating, while latter liners were replated with chromium only. Indications are that the channel-type chromium has an appreciably lower rate of wear than the pocket-

type chromium plating. Ultimate objective of project is to obtain a complete assembly on which relative wear rates of components are such that complete assembly will achieve maximum possible service life between scheduled overhauls, rather than to have one part of assembly reach its wear limit prematurely. Graphs, diagrams, photos.—INCO. 15283

## 8.9.5, 5.4.5, 4.6.11, 7.3

**Corrosion Prevention at the Stern Areas of H. M. Ships.** J. C. KINGCOMBE. *Corrosion Prevention & Control*, 4, No. 12, 35-40 (1957) Dec.

Stern area of a ship comprises propellers, their supporting brackets, rudder and area of hull in vicinity of these fittings. Propeller shaft, brackets and rudder are of steel, while propeller itself is normally made of bronze. So protection against galvanic corrosion has to be provided for the hull and steel fittings. To aggravate problem, shape of some of the fittings makes effective protection by coatings more difficult. Most serious form of deterioration, however, occurs in service resulting from motion of propellers in the sea. Turbulence set up around propellers is very severe and is capable of removing many of the toughest and most adherent coatings from adjacent areas and propeller itself may suffer cavitation attack. Prevention of corrosion is discussed as to cathodic protection in conjunction with neoprene on rudders and shaft brackets. Defective areas of rudders and brackets could be repaired by zinc spraying, following it with an overall coating of 6 thous. of zinc spray, and then neoprene or a coating of rubber cured in situ. Best single

contribution to solution would be to fabricate propeller in a material having little or no electrochemical effect on nearby metals and which is resistant to cavitation attack. Of metals, titanium shows some promise, but although it has good resistance to corrosion, much more requires to be done before it can be said that titanium propellers are answer to problem of corrosion at stern areas. Photos.—INCO. 14967

## 8.9.5, 6.4.2, 3.7.3

**Light Alloys in Shipbuilding.** J. E. TOMLINSON. *Metal Ind.*, 91, No. 16, 337-339 (1957) October 18.

Problems connected with the use of aluminum in shipbuilding are discussed. The choice of the alloy to be used is determined by the best compromise between mechanical strength and corrosion resistance under marine conditions. For plate and extrusion Lloyd and the British Standards specify aluminum-3.5 to 5.5% magnesium alloys, but for some other applications aluminum-magnesium-silicon alloys, together with cast alloys of the aluminum-magnesium and aluminum-silicon type are used. Fabrication methods are procedures recommended by the Aluminum Development Association are summarized. Nature and prevention of intercrystalline, stress and bimetallic corrosion are discussed. The problems in welding aluminum are classified as follows: (1) Chemical affinity of aluminum for oxygen, coupled with the closeness of the densities of aluminum and aluminum oxide, which introduce a risk of oxide inclusion; (2) Strong tendency of the metal to dissolve hydrogen, which introduces a risk of hydrogen porosity; (3) Possibility, with certain aluminum alloys, of cracking above the solids. Basic mechanisms are understood in all cases, and appropriate counter-measures have been devised and generally proved in practice.—ALL. 15103

## 8.10 Group 10

### 8.10.2, 8.9.4, 6.2.2

**Metallurgy Makes the Difference.** F. G. SEFING, INC. *Diesel Power*, 35, No. 11, 35-39 (1957) November.

Discussion of cases where metallurgy has met mounting challenges of increasing internal temperature and pressure in Diesel engines. Ring groove pounding and piston head burning can be prevented by mounting suitable inserts made of heat resistant high expansion metals such as Ni-Resist cast iron or 18-8 stainless. High expansion Ni-Resist inserts are commonly bonded to aluminum alloy pistons by the Al-Fin process and have proven excellent expedients to prevent aluminum piston failures. In some engines corrosive wear occurs in upper part of liners. Effects of frequent starts, corrosive fuels or other corrosive conditions can be prevented by use of Ni-Resist ductile iron. Use of high-strength cast irons as well as chromium plating for high surface hardness and superior wear has become common piston ring practice. Where manifold service demands high strength and toughness at 1200-1450 F, Ni-Resist alloys can be obtained in the magnesium treated or ductile variety. When high vibratory mechanical stresses are encountered or when heavy parts are mounted on manifold, one of the ductile Ni-Resist types with its high strength and toughness at service temperatures has withstood the service. Tables, photos.—INCO. 15087



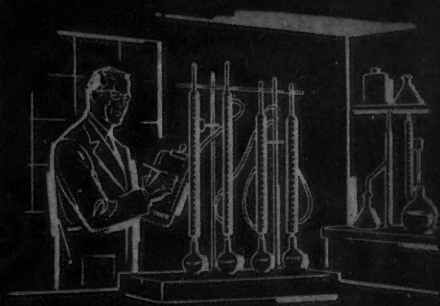
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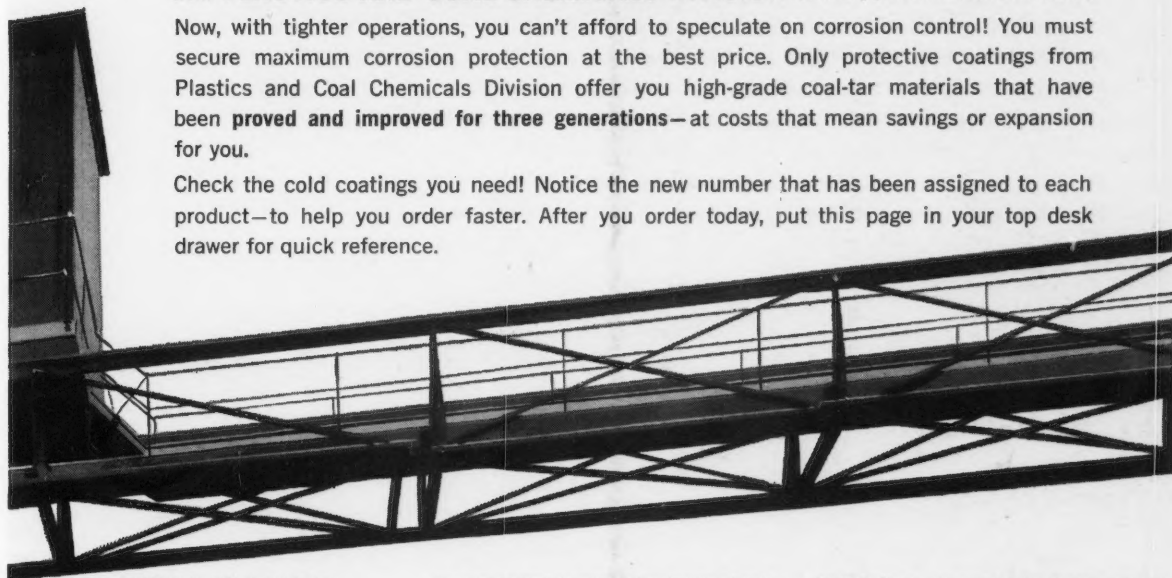


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- ☐ **Allied Chemical Coal-Tar Paint—(#112)**—A paint for all exposed metal work. Resists dilute nitric, sulfuric and hydrochloric acids, sulfur fumes and alkali. Use in presence of hydrogen sulfide—on pipelines, trestles, machinery, tanks, masonry and wood. In laundries, bleacheries, chemical, dye and steel plants, mines, bridges, etc. Brush or spray on. Two coats last five years under normal conditions. Covers 400 square feet per gallon per coat. Meets Military Specification MIL-P-6883.

- ☐ **Allied Chemical T.O.F. Solution—(#114)**—

Coal-tar material suitable for inside of potable water tanks and pipes, hydrants and hose houses and equipment where no residual taste or odor can be tolerated. Meets AWWA Specification D102-55T, Sec. 5.4.6. for "Cold-Applied T & O Tar Base Paint."

- ☐ **Allied Chemical Industrial Coating—(#120)**—Special purpose, reinforced, heavy-duty coal-tar pitch coating for severe corrosive conditions. Brush or spray it on metal, concrete and stone masonry. Reinforced with inert minerals. Equivalent to about three coats of ordinary paint. Withstands wet heat of 185° F. and dry heat of 400° F.

- ☐ **Allied Chemical 34Yc Coating—(#134)**—A plasticized thixotropic, coal-tar material that meets requirements of the U. S. Navy, Bureau of Yards and Docks Specification No. 34Yc. Use it on exposed metal work where corrosion is severe due to salt spray and other reactive conditions—piers, ships, floating drydocks, service vessels and for offshore oil and gas wells. Use also for nuclear

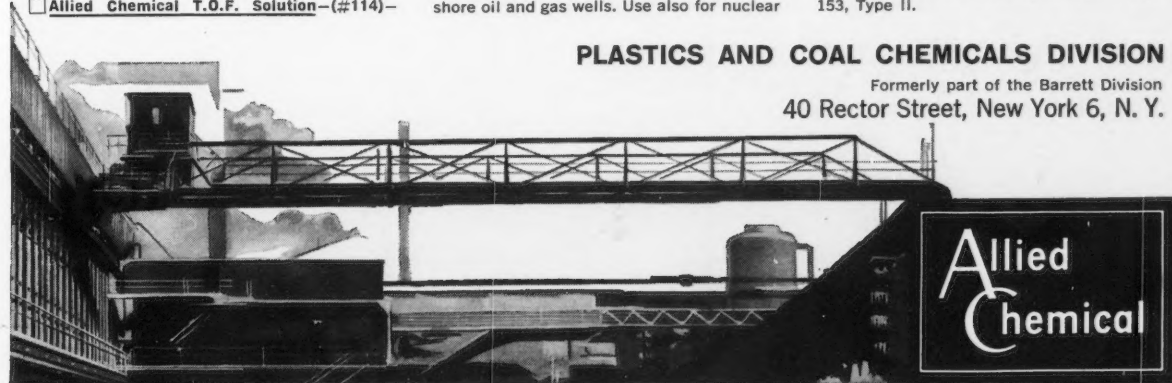
installations. Approved by Underwriters' Laboratories, Inc. for use on septic tanks—bituminous coated, System II.

- ☐ **Allied Chemical CA 50 Coating—(#150)**—A plasticized, gel-type, coal-tar material that meets U. S. Bureau of Reclamation Specification No. C.A. 50. Apply it at atmospheric temperatures, wherever extra-heavy and durable protection is required. Protects concrete work, dams, penstocks and bridge piers. Also, exposed trestle work, exposed concrete or metal work subject to extreme corrosion. Used when an engineer desires to achieve maximum thickness with minimum number of coats.

- ☐ **Allied Chemical Service Cement—(#160)**—Service mastic with high-grade, coal-tar pitch base, plastic material. Apply it cold to field joints of welded pipe. You don't have to prime or torch surface. Use it with our Pipeline Fabric. Inert, waterproof, non-corrosive to steel surfaces. Not affected by soil acids and alkalis. Meets Specification SSC-153, Type II.

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# A Review of the Phosphate Coatings

## Specified for the Protection of Metal Surfaces

By HUGH GEHMAN, Assistant Manager, Product Development Dept., AMCHEM PRODUCTS, INC.

Phosphate coatings are protective inorganic finishes that actually change the chemical nature of metal surfaces. The metal reacts with the applied phosphate solution to form a nonmetallic, crystalline coating which serves to:

- Improve paint adhesion
- Provide protection against corrosion
- Increase lubricity of friction surfaces
- Facilitate mechanical deformation of metals
- Decorate—in many instances

Satisfactory protection of steel, zinc and aluminum surfaces against corrosion, paint peeling and blistering, and hard wear requires precision methods of chemical conversion coating.

### Types of Conversion Coatings

There are seven classes of chemical conversion coatings commonly specified and used throughout industry today. They are as follows:

**Zinc-iron phosphate (ACP Granodine®).** This is the heaviest type of coating (gray in color) used for prepaint treatments on steel, iron and zinc surfaces. The process requires five or six operations: cleaning; rinsing; rust removal, if necessary; coating; rinsing; and a second rinse. Coating weight ranges from 100 to 600 mg per sq. ft.

Medium or large volume production of automobile bodies, appliances, projectiles and cabinets can be handled effectively.

The coating solution improves paint adhesion by forming a crystalline deposit over the metal surface. This deposit is rough, as revealed microscopically, and so offers an ideal gripping surface for paint particles.

**Manganese-iron phosphate (ACP Thermoil-Granodine®).** This is a heavy black coating used on friction surfaces to prevent galling, scoring and seizing of parts. Typical metal parts treated are pistons, piston rings, gears, cylinder liners, camshafts, tappets and various small arms components.

**Iron phosphate (ACP Duridine®).** This is a comparatively new process that places a light coating on surfaces for improved paint adhesion. Since cleaning and coating occur in the same bath, it has only three to five stages.

The iron phosphate treatment is a spray process suited for medium to large volume, large or small work. Pre-cleaning is normally unnecessary, an economy factor in its favor.

Products protected by this process are steel or iron fabricated units, such

as cabinets, washing machines and refrigerators. Weight of coating is 50 to 100 mg per sq. ft.

**Zinc phosphate (ACP Lithoform®).** This is a crystalline coating produced on galvanized iron and other zinc surfaces—also cadmium—for improving paint adhesion. The purpose of the coating is to provide a paint-gripping surface and to prevent the reaction between acidic components of the paint and the zinc metal, with the formation of soaps and loss of paint adhesion.

This coating is applied in weights of 75 to 500 mg per sq. ft. There are no limitations on volume or production or on size of products treated. Zinc phosphate coating is used on zinc alloy die castings, zinc or cadmium plated sheet or components, hot dip galvanized stock, and Galvanneal.

**Amorphous phosphate (ACP Alodine®).** This is a relatively new protective coating for aluminum and aluminum alloys. It may be used in place of anodic deposition for improved paint adhesion and corrosion resistance.

This coating is practical for production in any volume. Coating weight is 100 to 600 mg per sq. ft. Products treated include helmets, belt buckles, aircraft parts, bazookas and rocket motors, roofing and siding. Particularly good when aluminum is painted prior to forming.

**Zinc-iron phosphate for oil absorption (ACP Permadine®).** This is a relatively heavy coating adapted to the retention of rust-inhibiting drying or nondrying oils and waxes on ferrous metal surfaces. The coating is applied to a weight of 1000 to 4000 mg per sq. ft.

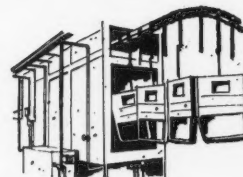
The process is satisfactory for large or small work in any volume—nuts, bolts, hardware, guns, tools, etc.

**Zinc-iron phosphate for metal forming (ACP Granodraw®).** This is a specialized coating used in conjunction with a suitable lubricant to facilitate the cold mechanical deformation of steel. The coating acts as an anchor for the lubricant throughout drawing, extrusion, and cold forming operations.

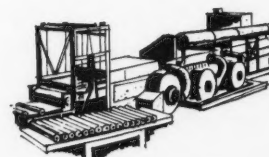
It is a successful treatment for products such as blanks and shells for cold forming, heavy stampings, impact extruded shapes, drawn wire and tube.

For more complete information about any one or all of these chemical conversion coatings, contact an ACP sales representative or write us at Ambler, Pa.

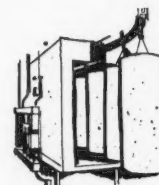
### Typical Installations of Phosphate Coating Systems



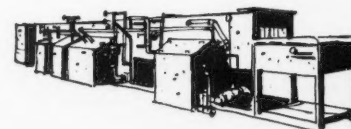
**Customer:** Truck manufacturer  
**Problem:** Preparing cab parts for painting  
**Cycle:** Phosphate wash; phosphate wash; rinse; chromic acid rinse; dry



**Customer:** Aluminum screen manufacturer  
**Problem:** Final finish of aluminum shade screen  
**Cycle:** Wash; rinse; phosphate coat; rinse; chromic acid rinse; dry



**Customer:** Water heater manufacturer  
**Problem:** Preparation of water heater shells for synthetic enameling  
**Cycle:** Phosphate wash; rinse; chromic acid rinse; dry



**Customer:** Hardware manufacturer  
**Problem:** Preparing hardware parts for painting  
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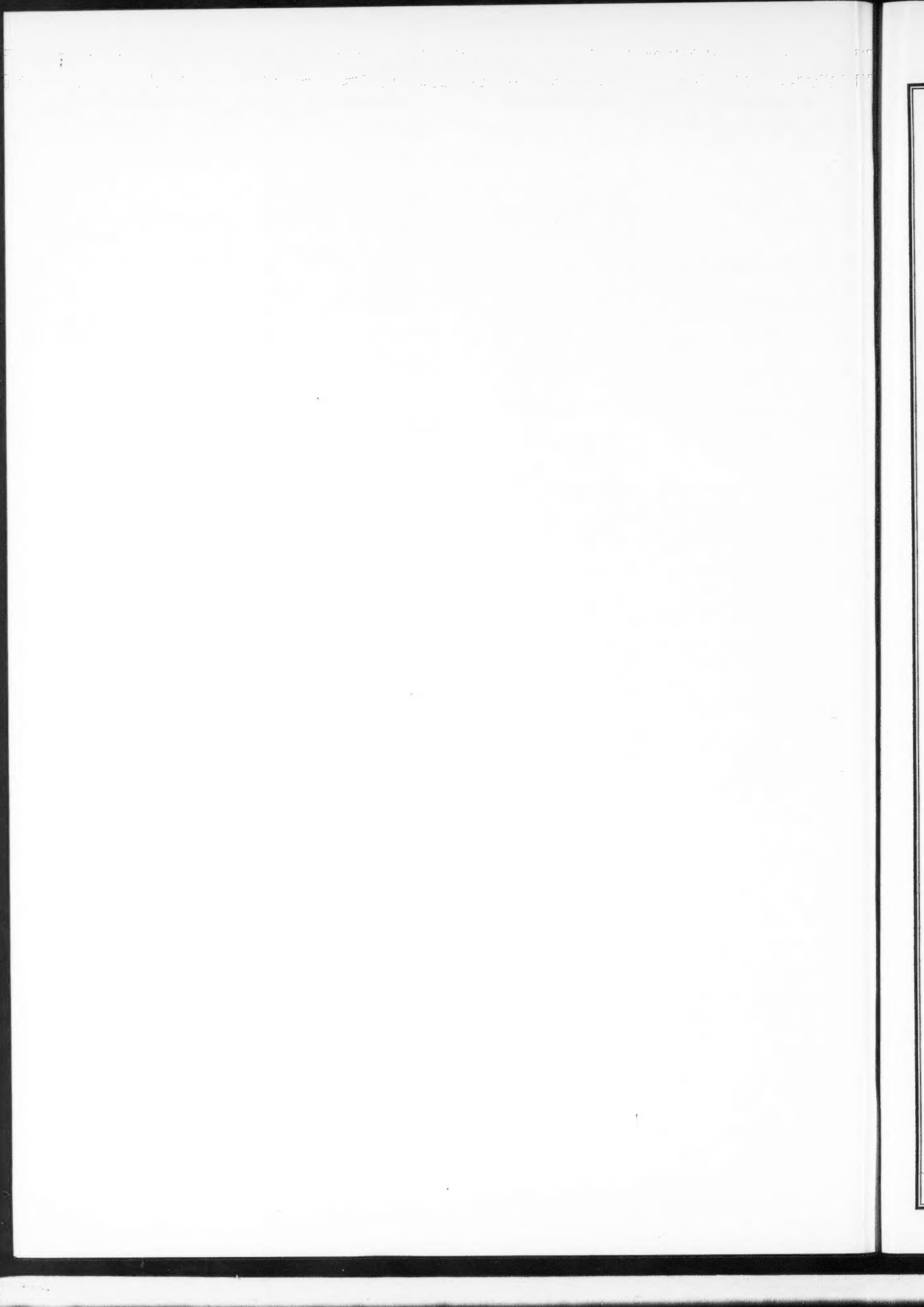
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# CORROSION ABSTRACTS

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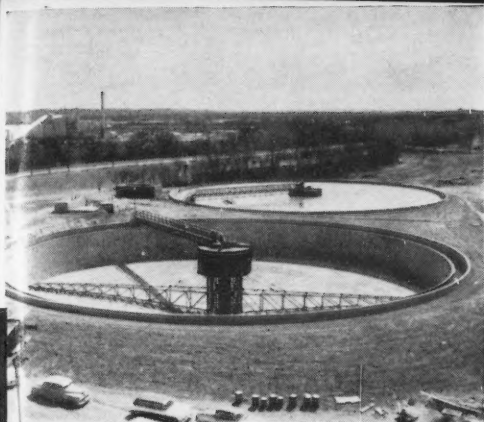
\* The following designations apply to the several groups listed under 8, INDUSTRIES:

1. Air conditioning, architecture and building, refrigeration, sewage and water.
2. Communications, power.
3. Agriculture, beverage, dairy, fermentation, food, sugar, starch.

4. Fuel, solids; fuel, gases; petroleum refining and production, rubber, atomic energy.
5. Ceramics, glass, pulp and paper, wood products.
6. Laundry soap and detergents, textile.
7. Graphic arts, instruments, jewelry, photography.

8. Chemical manufacturing, distilled liquor, electroplating, leather and tanning, metal fabrication and finishing, pharmaceuticals.
9. Aircraft, automotive, pipe line, railroad, shipping.
10. Explosives, metallurgy, mining ordnance and war materials, other.





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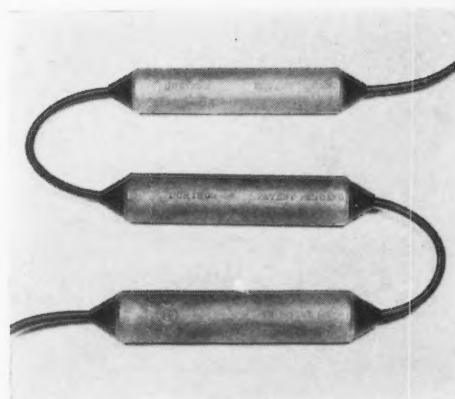
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**THE ANSWER:** Duriron impressed current anodes! Strings of Duriron 1 1/4" dia. x 9" long anodes were 1) extended along both sides of the rake arms, 2) hung vertically to protect the lower section influent wells and center structures, and 3) suspended horizontally on a 16 1/2' radius at a depth four feet below the water level.

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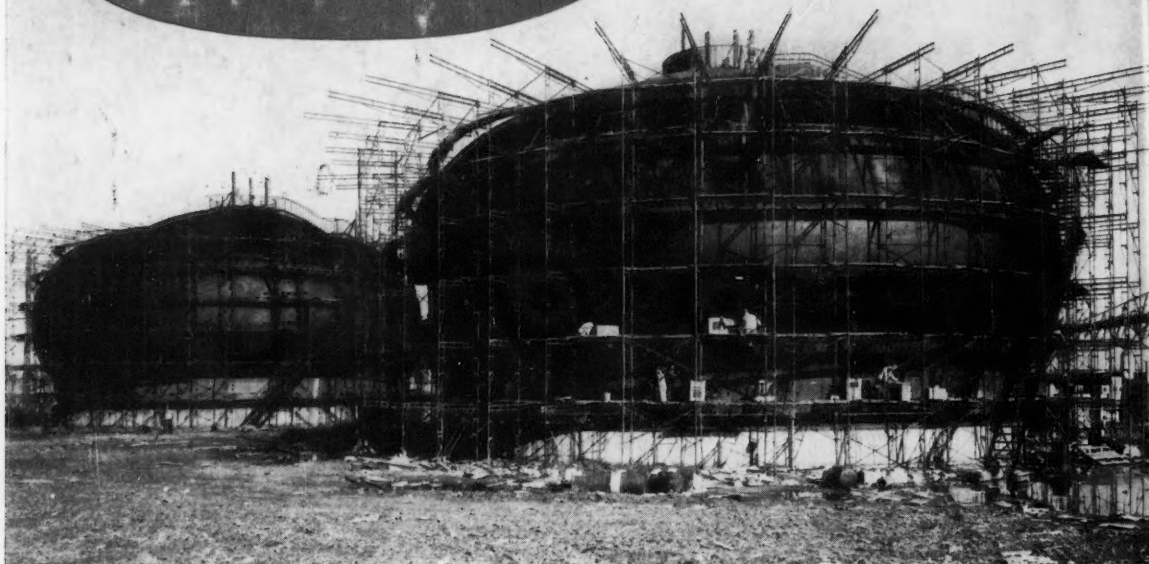
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PROVIDE SURE, LOW-COST  
CORROSION PROTECTION FOR  
BUTANE SPHEROIDS**



**Solution: Specify Time-Tested PITT CHEM *Insul-Mastic***

**S** ELECTION of a durable, heavy-duty moisture and vapor seal was an important consideration in planning the corrosion and insulation protection of five 40,000-barrel spheroid butane tanks at a large East Coast refinery.\*

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\*Name on request.

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